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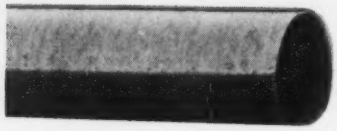

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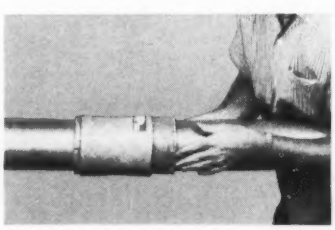

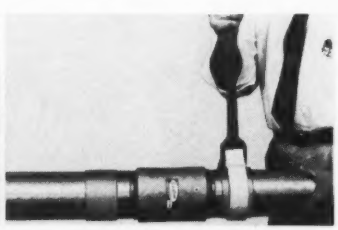

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THIS MONTH'S COVER—This is the air-conditioned Dallas Memorial Auditorium where the NACE 16th Annual Conference and 1960 Corrosion Show will be held. The show will be located in the ground floor exhibit space and meetings will be held elsewhere in the same building. There is parking space for 1100 cars.

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High Temperature, High Pressure Testing of Organic Coatings for Oil and Gas Well Tubing, by Cordell Garner

Solving Some Oil Ash Corrosion Problems, by R. S. Norris

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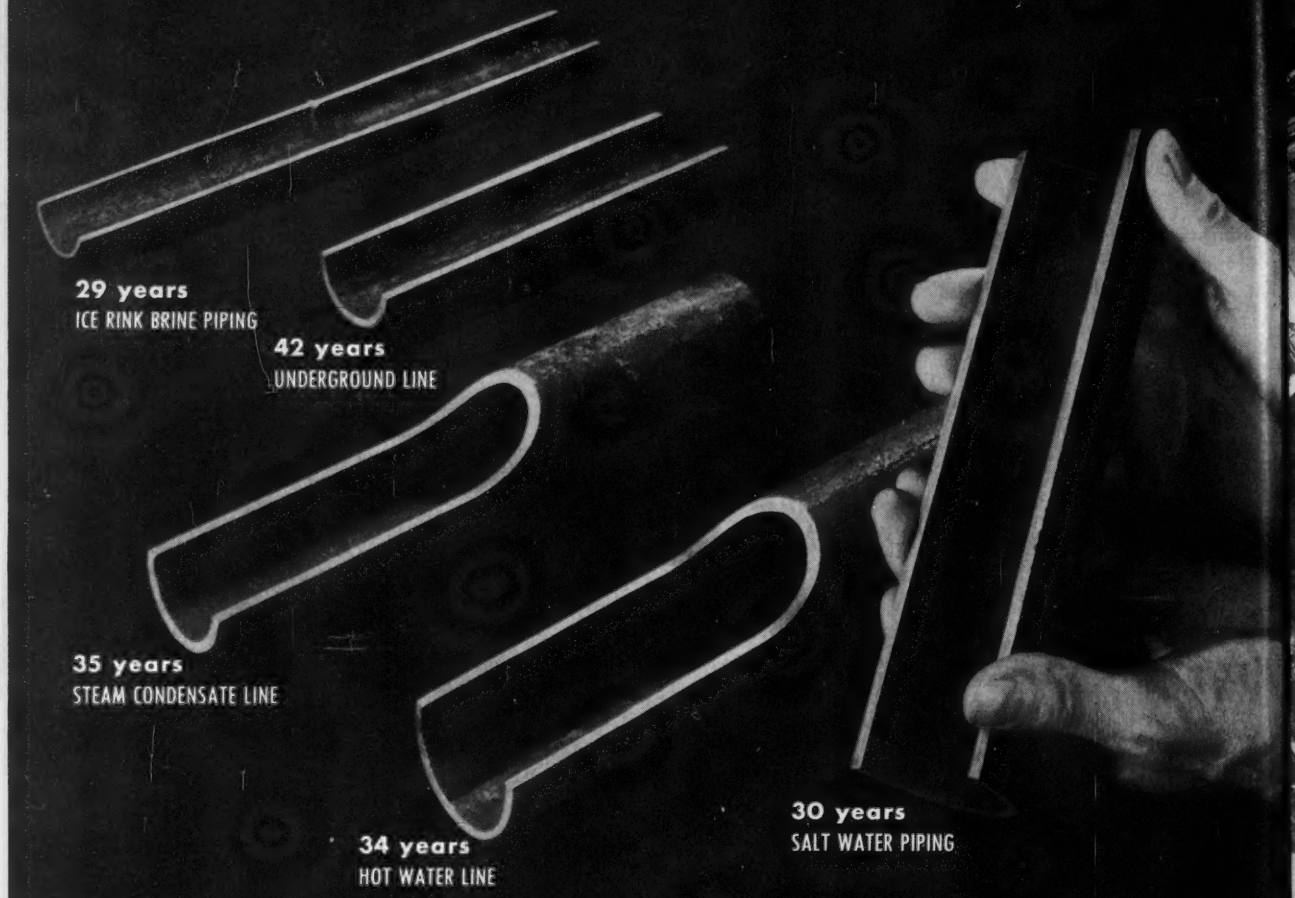

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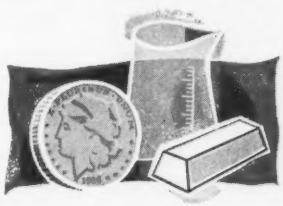

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T-3A-3 Materials Available for and Application of Corrosion Inhibitors

Lyle A. Timm, Chairman; 8450 Ferry Road, Gross Ile, Michigan

T-3B Corrosion Products

A. H. Roebuck, Chairman; The Western Co., Midland Tower Bldg., P. O. Box 310, Midland, Texas

D. A. Vaughan, Vice Chairman, Battelle Memorial Institute, 505 King Avenue, Columbus, Ohio.

T-3C Annual Losses Due to Corrosion

S. K. Coburn, Chairman, Association of American Railroads, 3140 South Federal Street, Chicago 16, Illinois

T-3D Instruments for Measuring Corrosion

E. H. Thalmann, Chairman; Ebasco Services, Inc., 2 Rector St., New York, N. Y.

F. W. Ringer, Vice Chairman; 7 Hampden Ave., Narberth, Pa.

T-3D-1 Electrical Holiday Inspection of Coatings

W. A. Broome, Chairman; Arkansas-Louisiana Gas Co., Box 1734, Shreveport, La.

T-3E Railroads

L. J. Nicholas, Chairman; The Pullman Co., Engineer of Research, Room 5-40, Merchandise Mart Plaza, Chicago, Ill.

G. M. Magee, Vice Chairman, Assoc. American Railroads, 3140 South Federal Street, Chicago, Ill.

T-3E-1 Corrosion of Railroad Tank Cars

J. R. Spraul, Chairman; General American Transportation Corp., 150 West 151st Street, East Chicago, Ind.

C. M. Jekot, Vice Chairman; DeSoto Paint & Varnish Co., P. O. Box 186, Garland, Texas.

L. R. Honnaker, Vice Chairman, Engineering Materials Group, Eng. Dept., E. I. du Pont de Nemours & Co., Inc., 13W15 Louviers Bldg., Wilmington, Del.

T-3E-2 Corrosion in Railroad Hopper Cars

C. L. Crockett, Chairman; Norfolk and Western Railway, Motive Power Dept., Roanoke, Va.

T-3F Corrosion by High Purity Water

R. U. Blaser, Chairman; Research Center, Babcock & Wilcox Co., Box 835, Alliance, Ohio.

M. G. Bloom, Vice Chairman; Metallurgy Div., Naval Research Lab., Washington, D. C.

T-3F-2 Inhibitors

R. C. Ulmer, Chairman, Combustion Engineering Co., 200 Madison Avenue, New York, N. Y.

T-3F-3 Corrosion Products

W. L. Pearl, Chairman, General Electric Company, Atomic Power Equipment Dept., Box 254, San Jose, Calif.

T-3F-6 Intercommittee Activities

E. P. Partridge, Chairman, Hall Labs., Box 1346, Pittsburgh, Pa.

T-3F-7 Bibliography

W. K. Boyd, Chairman; Battelle Memorial Institute, 505 King Ave., Columbus, Ohio

T-3G Cathodic Protection

Dr. T. P. May, International Nickel Co., Inc., P. O. Box 262, Wrightsville Beach, North Carolina

T-3G-1 Cathodic Protection of Hull Bottoms of Ships

L. P. Sudrabin, Chairman, Electro Rust-Proofing Corp., Box 178, Newark, New Jersey.

T-3G-2 Cathodic Protection of Heat Exchangers

R. B. Teel, Chairman, The International Nickel Co., Inc., Box 262, Wrightsville Beach, N. C.

T-3G-3 Cathodic Protection of Process Equipment

A. A. Brouwer, Chairman, The Dow Chemical Co., Midland, Michigan.

T-3H Tanker Corrosion

W. S. Quimby, Chairman; Res. & Tech. Dept., The Texas Company, Box 509, Beacon, New York.

T4 Utilities

D. R. Werner, Chairman; American Tel. & Tel. Co., Room 1701, 324 East 11th St., Kansas City 6, Missouri.

J. B. Prime, Jr., Vice Chairman; Florida Power & Light Co., P. O. Box 3100, Miami 30, Florida

T-4A Effects of Electrical Grounding on Corrosion

T. R. Stilley, Chairman; Good-All Electric Mfg. Co., Good-All Bldg., Ogallala, Neb.

O. W. Zastrow, Vice Chairman; Electric Engineering Div., Rural Electrification Adm., U.S.D.A., Washington 25, D. C.

T-4A-3 Methods and Materials for Grounding

I. C. Dietze, Chairman; Dept. of Water & Power, Box 3669, Terminal Annex, Los Angeles 54, Cal.

T-4B Corrosion of Cable Sheaths

T. J. Maitland, Chairman, American Telephone & Telegraph Co., 32 Avenue of the Americas, New York, N. Y.

H. M. Clayton, Vice Chairman; Memphis Gas, Light and Water Div., Box 388, Memphis, Tenn.

T-4B-1 Lead and Other Metallic Sheaths

T. J. Maitland, Chairman, American Telephone & Telegraph Co., 32 Avenue of the Americas, New York 13, New York.

T-4B-2 Cathodic Protection

J. J. Pokorny, Chairman; Cleveland Electric Illuminating Co., P. O. Box 5000, Cleveland 1, Ohio.

T-4B-3 Tests and Surveys

D. R. Werner, Chairman, American Telephone & Telegraph Co., 324 East 11th Street, Room 1701, Kansas City, Mo.

H. M. Clayton, Vice Chairman, Memphis Gas, Light and Water Div., Box 388, Memphis, Tenn.

T-4B-4 Protection of Pipe Type Cables

H. W. Dieck, Chairman, Long Island Lighting Company, 175 Old Country Road, Hicksville, New York.

J. B. Prime, Jr., Vice Chairman, Florida Power & Light Co., Box 3100, Miami, Florida.

T-4B-5 Non-Metallic Sheaths and Coatings

C. L. Mercer, Chairman; Southwestern Bell Tel. Co., Box 58, Westfield, Texas

T-4B-6 Stray Current Electrolysis

J. Svetlik, Chairman, Northern Indiana Public Service Co., 5265 Hohman Ave., Hammond, Indiana.

G. H. Cantwell, Vice Chairman, Indiana Bell Telephone Co., 240 N. Meridian St., Indianapolis, Indiana.

T-4D Corrosion by Deicing Salts

W. H. Bruckner, Chairman; University of Illinois, Urbana, Illinois.

K. G. Compton, Vice Chairman; Bell Telephone Laboratories, Murray Hill, New Jersey.

T-4D-1 Procedures for Conducting Field Tests Below Ground

F. E. Kulman, Chairman, Consolidated Edison Co. of New York, Inc., 4 Irving Place, New York, N. Y.

T-4D-2 Coordination of Field Programs Between Cities and Transportation Companies

George Illig, Chairman, Calgon, Inc., 323 Fourth Ave., Pittsburgh, Penn.

T-4E Corrosion by Domestic Waters

T. E. Larson, Chairman, Illinois State Water Survey, Box 232, Urbana, Illinois.

J. F. J. Thomas, Vice Chairman, Industrial Water Section; Dept. of Mines & Tech. Surveys; 40 Lydia Street, Ottawa, Ontario, Canada

T-4E-1 Hot Water Tank Corrosion

R. C. Weast, Chairman; Associate Professor of Chemistry, Case Institute of Technology, University Circle, Cleveland 6, Ohio.

T-4F Materials Selection for Corrosion Mitigation in the Utility Industry

W. T. Burgess, Chairman; Metropolitan Utilities District, 18th & Harney Street, Omaha 2, Nebraska

I. C. Dietze, Vice Chairman; Dept. of Water & Power, Box 3669, Terminal Annex., Los Angeles 54, California.

T-4F-1 Materials Selection in the Water Industry

Daniel Cushing, Chairman, 148 State St., Boston, Massachusetts.

Chester Anderson, Vice Chairman; The Crane Co., 836 South Michigan Avenue, Chicago 5, Ill.

Peter Kahn, Secretary; Metcalf & Eddy, 1300 Statler Bldg., Boston, Mass.

T-4F-2 Materials Selection in the Electric Industry

F. E. Kulman, Chairman; Consolidated Edison Co. of New York, Inc., 4 Irving Place, New York 3, New York

T5 Corrosion Problems in the Process Industries

C. P. Dillon, Chairman; Union Carbide Chemicals Co., Div. of Union Carbide Corp., P. O. Box 2831, Charleston, W. Virginia

T-5A Corrosion in Chemical Processes

C. P. Dillon, Chairman; Union Carbide Chemicals Co., Div. of Union Carbide Corp., P. O. Box 2831, Charleston, W. Va.

W. H. Burton, Vice-Chairman, General Chemical Division, Allied Chemical & Dye Corp., Camden 3, New Jersey

T-5A-1 Sulfuric Acid

W. A. Luce, Chairman, The Duriron Co., Box 1019, Dayton, Ohio.

T-5A-3 Acetic Acid

J. A. Manning, Jr., Chairman; Celanese Corp. of America, Box 1414, Charlotte, N. C.

T-5A-4 Chlorine

L. W. Gleekman, Chairman; Wyandotte Chemicals Corp., 1609 Biddle Ave., Wyandotte, Mich.

G. F. Walther, Vice Chairman; Westvaco Chemical Div., Food Machinery & Chem. Corp., Box 8127, South Charleston, W. Va.

T-5A-5 Nitric Acid

N. D. Groves, Chairman; Apartment 33-B, Brookline Manor, Reading, Pa.

F. H. Meyer, Vice Chairman; National Lead Co. of Ohio, Box 158, Mt. Healthy Station, Cincinnati 31, Ohio.

T-5A-6 HF Corrosion

T. L. Hoffman, Chairman, Phillips Petroleum Co., Atomic Energy Div., Box 1259, Idaho Falls, Idaho.

F. H. Meyer, Jr., Vice Chairman; National Lead Co. of Ohio, Box 158, Mt. Healthy Station, Cincinnati 31, Ohio

T. F. Degnan, Secretary; 701 W. 22nd Street, Wilmington 2, Delaware

T-5B High Temperature Corrosion

R. T. Foley, Chairman; General Electric Co., General Eng. Lab., Room 2004, Bldg. 37, Schenectady, New York.

David Roller, Vice-Chairman; 5844 Access Drive, Dayton 3, Ohio.

T-5C Corrosion by Cooling Waters

T-5C-1 Corrosion by Cooling Waters (South Central Region)

J. M. Brooke, Chairman; Phillips Petroleum Co., Sweeny Refinery, Sweeny, Texas

T-5D Plastic Materials of Construction

R. E. Gackenbach, Chairman; American Cyanamid Co., Organic Chemical Div., Bound Brook, N. J.

O. H. Fenner, Vice-Chairman; Monsanto Chemical Co., 1700 South Second St., St. Louis 4, Mo.

T-5D-1 Questionnaires

S. W. McIlrath, Chairman. 151 E. 214th Street, Euclid 23, Ohio.

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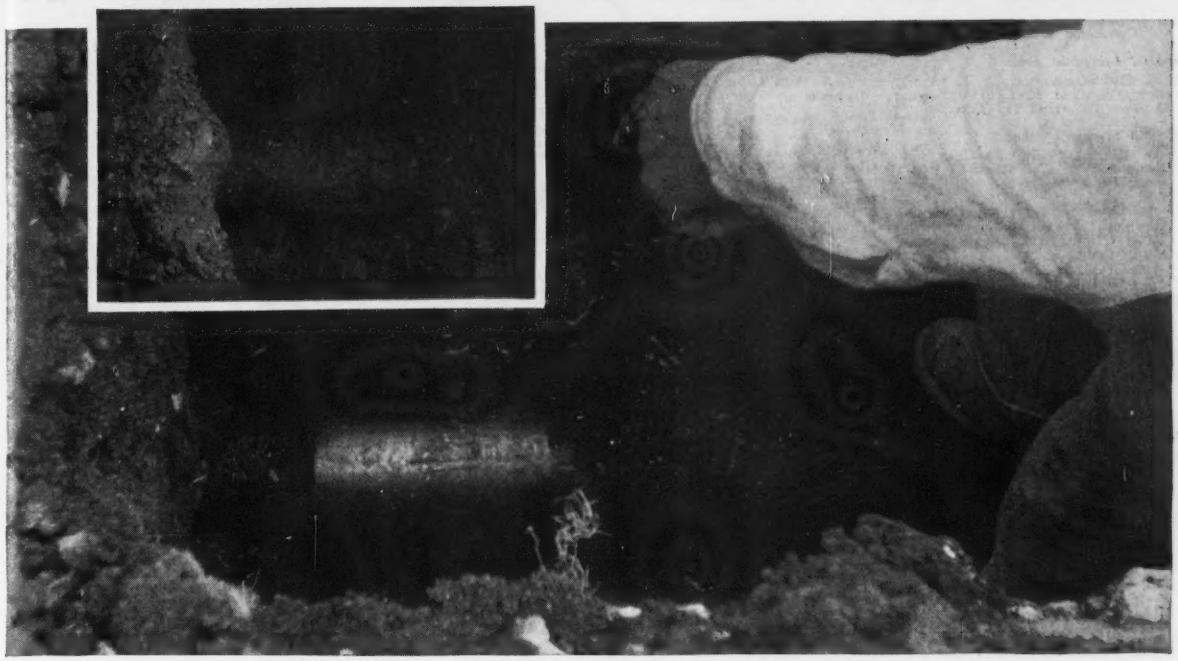

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T-5D-2 Inorganic Acids

R. J. Collins, Secretary; B. F. Goodrich Chemical Co., 3135 Euclid Avenue, Cleveland 15, Ohio

T-5D-3 Inorganic Alkalies

Peter Kimen, Chairman, Champion Paper & Fibre Co., Box 872, Pasadena, Texas.

L. B. Connelly, Secretary, Eastman Chemical Products Company, 704 Texas National Bank Building, Houston 2, Texas.

T-5D-4 Gases

Beaumont Thomas, Chairman, Stebbins Eng. & Mfg. Co., Eastern Blvd., Watertown, New York.

T-5D-5 Water and Salt Solutions

Paul Elliott, Chairman, Naugatuck Chemical Company, Kralastic Development, Naugatuck, Conn.

T-5D-6 Organic Chemicals

Wade Wolfe, Jr., Secretary, 73 Ermann Drive, Buffalo 17, New York.

T-5D-7 Engineering Design

O. H. Fenner, Chairman, Monsanto Chemical Company, 1700 South Second Street, St. Louis 4, Missouri.

K. A. Phillips, Vice-Chairman, American Zinc, Lead & Smelting Co., P. O. Box 495, East St. Louis, Illinois.

T-5D-8 Methods and Criteria for Evaluating Plastics in Chemical Environment

R. F. Clarkson, Chairman, Olin Mathieson Chemical Corp., Mathieson Building, Baltimore, Md.

T-5E Stress Corrosion Cracking of Austenitic Stainless Steel

L. Miller Rogers, Chairman; Union Carbide Chemicals Co., Box 471, Texas City, Texas.

E. V. Kunkel, Vice Chairman; Celanese Corp., Box 148, Bishop, Texas.

T6 Protective Coatings

L. S. Van Delinder, Chairman; Union Carbide Chemicals Co., Development Dept., South Charleston, W. Va.

O. H. Fenner, Vice-Chairman; Monsanto Chemical Co., 1700 South Second St., St. Louis 4, Mo.

T-6A Organic Coatings and Linings for Resistance to Chemical Corrosion

C. G. Munger, Chairman; Amercoat Corp., 4809 Firestone Blvd., South Gate, Cal.

L. S. Van Delinder, Vice Chairman; Union Carbide Chemicals Co., Development Dept., South Charleston, W. Va.

J. I. Richardson, Secretary; Amercoat Corp., 4809 Firestone Blvd., South Gate, Cal.

T-6A-1 Rubber Linings

R. McFarland, Jr., Chairman; Hills-McCanna Co., 400 Maple Street, Carpentersville, Illinois

T-6A-2 Vinyl Coatings

T-6A-3 Vinylidene Chloride Polymers

R. L. Brown, Chairman, The Dow Chemical Co., Midland, Mich.

T-6A-4 Phenolics

Forest Baskett, Chairman; Sheet Metal Engineers, Inc., Box 9384, Houston 11, Texas

T-6A-5 Polyethylene

L. S. Van Delinder, Chairman, Carbide & Carbon Chemicals Co., South Charleston, W. Va.

T-6A-6 Neoprene

J. R. Galloway, Chairman; E. I. du Pont de Nemours & Co., Inc., 2601A West Grove Lane, Houston 6, Texas

T-6A-7 Thermosetting Coal Tar Coatings

Woodrow E. Kemp, Chairman; Koppers Co., Inc., 15 Plum Street, Verona, Pa.

T-6A-8 Methacrylates

T-6A-9 Furanes

T-6A-10 Polyesters

D. D. Cone, Chairman; c/o Sunrise Coffee Shop, 118 East Texas, Baytown, Texas

T-6A-11 Epoxys

C. G. Munger, Chairman, Amercoat Corp., 4809 Firestone Blvd., South Gate, Cal.

T-6A-12 Fluorocarbons

T-6A-13 Chlorinated Rubbers

Appointment Pending

T-6A-14 Organic-Brick Covered

T-6A-15 Rigid Vinyls

C. G. Munger, Chairman; Amercoat Corp., 4809 Firestone Blvd., Southgate, Calif.

T-6A-16 Coal Tar Coatings

T-6A-17 Polyurethanes

Richard Sansone, Chairman; Mobay Chemical Company, 1815 Washington Road, Pittsburgh 34, Pa.

T-6A-18 Hypalon

J. R. Galloway, Chairman; E. I. du Pont de Nemours & Co., Inc., 2601A West Grove Lane, Houston 6, Texas

T-6A-19 Asphalts

T-6A-20 Plastisols

O. H. Fenner, Chairman; Monsanto Chemical Co., 1700 South Second St., St. Louis 4, Mo.

T-6B Protective Coatings for Resistance to Atmospheric Corrosion

Robert P. Suman, Chairman; Pittsburgh Plate Glass Co., P. O. Box 21114, Houston, Texas

M. W. Belue, Jr., Vice Chairman; Champion Paper Fibre Co., P. O. Box 872, Pasadena, Texas

T-6B-1 Linseed and Other Drying Oils

T-6B-2 Ester Gum Oil

R. L. Liston, Chairman, Cook Paint & Varnish Co., Box 3088, Houston, Tex.

T-6B-3 Straight Phenolic Oil Varnish

John W. Nee, Chairman, Briner Paint Mfg. Co., 3713 Agnes St., Corpus Christi, Texas.

T-6B-4 Modified Phenolic Oil Varnish

John W. Nee, Chairman.

T-6B-5 Straight Alkyd Varnish

T-6B-6 Modified Alkyd Varnish

T-6B-7 Epoxy Esters

T-6B-8 Epoxy (Amine Cured)

T-6B-9 Chlorinated Rubber

Otto Grosz, Chairman, The California Co., Box 128, Harvey, La.

T-6B-10 Vinyls

John I. Richardson, Chairman; Amercoat Corporation, 4809 Firestone Blvd., South Gate, Cal.

T-6B-11 Metallic Silicates

T-6B-12 Coal Tar

M. Mitchell, Chairman, Reilly Tar & Chem. Co., 1616 Merchants Bank Bldg., Indianapolis, Ind.

T-6B-13 Asphalt

T-6B-14 New Developments

T-6B-15 Silicones

W. F. Gross, Chairman; c/o Norton Jaggard, Manager Engineering, Arabian American Oil Co., 505 Park Ave., New York 22, N. Y.

T-6C Protective Coatings for Resistance to Marine Corrosion

John F. Oliveira, Chairman, Amercoat Corporation, 2908 Knoll Acres Drive, Baltimore 34, Md.

T-6D Industrial Maintenance Painting

R. H. Bacon, Chairman; Dow Chemical Co., Maintenance Shops, Freeport, Texas

C. R. Martinson, Vice Chairman; Monsanto Chemical Company, 1700 South Second Street, St. Louis, Mo.

T-6D-1 Economics of Maintenance Painting

S. L. Lopata, Chairman, Carboline Co., 331 Thornton Ave., St. Louis, Mo.

T-6D-2 Standardization of Scope of Painting Specifications

L. L. Sline, Chairman, Sline Industrial Painters, 2162 Gulf Terminal Dr., Houston, Tex.

T-6D-3 Paint Programs

F. Parker Helms, Chairman; Union Carbide Chemicals Co., Texas City, Texas

T-6D-4 Specifications for Shop Cleaning & Priming

L. L. Sline, Chairman; Sline Industrial Painters, 2162 Gulf Terminal Drive, Houston 23, Texas.

T-6D-5 Exploratory Task Force on Painter Safety & Painter Education

(Officers to be appointed.)

T-6E Protective Coatings in Petroleum Production

C. G. Fritts, Chairman; Socony Paint Products Co., Box 1740, Houston, Texas.

F. E. Blount, Vice Chairman; Magnolia Petroleum Co., Box 900, Dallas, Texas.

T-6F Protective Interior Linings, Application and Methods

W. P. Cathcart, Chairman, Tank Lining Corporation, 246 Washington Road, Pittsburgh, Pennsylvania.

A. R. Gabel, Vice-Chairman; The Dow Chemical Co., Midland, Mich.

J. H. Cogshall, Secretary; Pennsalt Chemicals Corp., Corrosion Engineering Products Dept., Natrona, Pa.

T-6F-1 Curing

K. G. Lefevre, Chairman; Metalweld, Inc., 2617 Hunting Park Ave., Philadelphia, Pa.

T-6F-2 Surface Preparation

T-6F-3 Inspection

J. L. Barker, Chairman; Union Carbide Chemicals Co., Div. of Union Carbide Corp., P. O. Box 8004, Bldg., 82-421, South Charleston 3, W. Va.

T-6F-4 Safety

J. L. Barker, Temporary Chairman; Union Carbide Chemicals Co., Div. of Union Carbide Corp., P. O. Box 8004, Bldg. 82-421, South Charleston 3, W. Va.

T-6F-5 Application

T-6G Surface Preparation for Organic Coatings

Joseph Bigos, Temporary Chairman; United States Steel Corp., Applied Research Lab., Monroeville, Pa.

T-6H Glass Linings and Vitreous Enamels

R. McFarland, Jr., Vice Chairman; Hills-McCanna Co., 400 Maple Street, Carpentersville, Illinois

T-6J Protective Coating Application Problems

T-6J Los Angeles Area, Protective Coating Application Problems

Fred M. McConnell, Chairman, Service Coatings Corp., P. O. Box 524, Wilmington, Cal.

Newell W. Tune, Vice-Chairman, L. A. Water & Power, 510 East Second St., Los Angeles, Cal.

W. M. Jakway, Secretary, Bechtel Corporation, P. O. Box 58424, Los Angeles 58, Cal.

Paul C. Briley, Assistant Secretary, National Lead Co., 3113 East 26th St., Los Angeles, Cal.

T-6J-1 Specification Writing

Newell Tune, Chairman, Dept. of Water & Power, 510 East Second St., Los Angeles, Calif.

T-6J-2 Application Procedure

T-6J-3 Inspection Techniques

Miss Flora L. Davis, Chairman; Hughes Aircraft, Bldg. #20, Mail Station 1309, Florence and Teale, Culver City, California

T-6J-4 Cost Evaluation

LeRoy F. Doyle, Chairman, Magna Products Inc., 11808 Bloomfield Avenue, Santa Fe Springs, Cal.

T-6K Corrosion Resistant Construction With Masonry and Allied Materials

L. R. Honnaker, Chairman; E. I. du Pont de Nemours & Co., Inc., Eng. Dept., 33E20 Louviers Bldg., Wilmington, Del.

T-6R Protective Coatings Research

J. H. Cogshall, Chairman; Pennsalt Chemicals Corp., Corrosion Engineering Products Dept., Natrona, Pa.

T7 Corrosion Coordinating Committee

C. L. Mercer, Chairman; Southwestern Bell Tel. Co., P. O. Box 58, Westfield, Texas

T-7A Northeast Region Corrosion Coordinating Committee

C. A. Erickson, Jr., Chairman; The Peoples Natural Gas Co., Two Gateway Center, Pittsburgh 22, Pa.

L. Andrew Kellogg, Vice-Chairman; Niagara Mohawk Power Corp., 300 Erie Blvd., West Syracuse, N. Y.

New Jersey Committee on Corrosion

T. S. Watson, Chairman; Socony Mobil Oil Co., Inc., Eastern Pipe Lines Div., P. O. Box 989, Plainfield, N. J.

P. S. McKenna, Vice Chairman; Delaware Lackawanna & Western Railroad, Hoboken, N. J.

R. J. Bishop, Secretary; Public Service Electric & Gas Co. of N. J., 80 Park Place, Newark, N. J.

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Joseph Moriarty, Vice-Chairman; New York Telephone Co., New York, N. Y.

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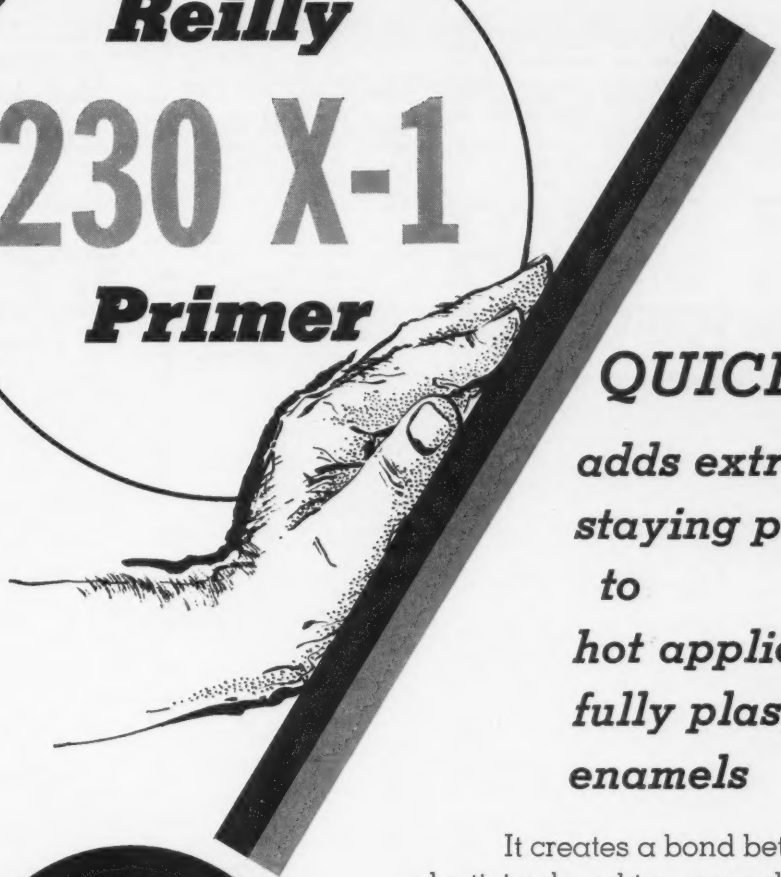

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R. L. Seifert, Vice-Chairman, Tennessee Gas Transmission Company, P. O. Box 98, Hamburg, New York.

E. L. Varney, Secretary, Iroquois Gas Corporation, 365 Mineral Springs Road, Buffalo, New York

Southern W. Virginia Corrosion Coordinating Committee

K. R. Gosnell, Chairman; Development Dept., Union Carbide Chemicals Co., South Charleston, West Virginia

P. F. Sweeney, Vice Chairman; Chesapeake & Potomac Tel. Co. of W Va., 816 Lee St., Charleston 5, W. Virginia

James A. Parker, Secretary; United Fuel Gas Co., P. O. Box 1273, Charleston 25, W. Virginia

Greater Boston Electrolysis Committee

William Helm, Jr., Chairman; Boston Gas Co., 100 Arlington St., Boston, Mass.

J. J. Molloy, Vice Chairman; Cambridge Electric Light Co., 46 Blackstone St., Cambridge, Mass.

Henry E. Wilkins, Secretary-Treasurer; Central Laboratory, New England Electric System, 939 Southbridge Street, Worcester 10, Mass.

Western Pennsylvania Corrosion Committee

R. M. Keller, Chairman; Atlantic Pipe Line Co., 260 S. Broad St., Philadelphia 1, Pa.

G. C. Picht, Jr., Vice Chairman; American Tel. & Tel. Co., 429 Fourth Avenue, Pittsburgh 19, Pa.

John B. Vrable, Secretary; New York State Natural Gas Corp., Two Gateway Center, Pittsburgh 22, Pa.

Pittsburgh Corrosion Committee

C. M. Rutter, Chairman; Equitable Gas Co., 420 Blvd. of the Allies, Pittsburgh, Pa.

D. W. Kissinger, Vice Chairman; Duquesne Light Co., 1241 Reedsdale Street, Pittsburgh 33, Pa.

E. W. Steel, Secretary, The Bell Telephone Co. of Pa., 416 Seventh Ave., Pittsburgh 19, Pa.

T-7B North Central Region Corrosion Coordinating Committee

Paul C. Hoy, Chairman; Dayton Power & Light Co., 25 N. Main St., Dayton, Ohio.

J. O. Mandley, Vice Chairman, Michigan Consolidated Gas Co., 415 Clifford, Detroit, Mich.

Chicago Area Committee on Underground Corrosion

L. E. Nichols, Chairman; Corrosion Engineer, Northern Illinois Gas Co., 615 Eastern Ave., Bellwood, Ill.

J. Svetlik, Vice-Chairman; Senior Electrolysis Engineer, Northern Indiana Public Service Co., 5265 Hohman Ave., Hammond, Ind.

M. D. Fletcher, Secretary; Illinois Bell Telephone Co., Midstate Division, 215 W. Randolph St., 7th Floor, Chicago, Ill.

Columbus and Central Ohio Committee on Corrosion

N. R. Patton, Chairman; Columbus & Southern Ohio Electric Co., 215 North Front St., Columbus 15, Ohio

C. H. Bope, Secretary, Columbus & Southern Ohio Electric Co., 215 North Front St., Columbus 15, Ohio

Toledo and Northwestern Ohio Committee on Corrosion

L. H. West, Chairman; 339 W. Fremont St., Fostoria, Ohio

O. J. Mauter, Vice Chairman; Ohio Bell Tel. Co., 121 Huron Street, Toledo 4, Ohio

I. H. Heitkamp, Secretary; Toledo Edison Co., 420 Madison Avenue, Toledo 4, Ohio

T-7C Southeast Region Corrosion Coordinating Committee

Ernest W. Seay, Jr., Chairman, Chesapeake & Potomac Tel. Co. of Va., 120 W. Bute St., Norfolk, Va.

R. W. Hamel, Vice-Chairman; Commonwealth Natural Gas Corp., Box 2350, Richmond, Va.

The Louisville (Ky.) Joint Electrolysis Committee

Stuart H. Gates, Chairman, Southern Bell Tel. & Tel. Co., Louisville, Ky.

John L. Gray, Vice-Chairman, Louisville Gas & Electric Co., Louisville, Ky.

Walter Zell, Secretary, Louisville Gas & Electric Co., Louisville, Ky.

T-7D South Central Region Corrosion Coordinating Committee

C. L. Mercer, Chairman; Southwestern Bell Tel. Co., Box 58, Westfield, Texas

C. L. Woody, Vice Chairman, United Gas Corporation, Box 2628, Houston, Texas.

Houston Corrosion Coordinating Committee

C. L. Mercer, Chairman, Southwestern Bell Telephone Co., Box 58, Westfield, Texas.

San Antonio Area Corrosion Committee

Philip S. Donnell, Chairman; City Water Board, P. O. Box 2449, San Antonio 6, Texas

Carl M. Thorn, Vice Chairman; Southwestern Bell Tel. Co., P. O. Box 2540, San Antonio 6, Texas

I. A. Flodin, Secretary; City Water Board, P. O. Box 2449, San Antonio 6, Texas

T-7E Western Region Corrosion Coordinating Committee

Irwin C. Dietze, Chairman, Dept. of Water & Power, Box 3669, Terminal Annex, Los Angeles, Calif.

J. S. Dorsey, Vice-Chairman; Southern California Gas Co., Box 3249, Terminal Annex, Los Angeles 54, Cal.

Central California Cathodic Protection Committee

R. M. Evans, Chairman; Standard Oil Co., 11-C Camp, Taft, Cal.

J. W. Ritter, Vice Chairman; Southern California Gas Co., Box T, Taft, Cal.

J. R. Bryant, Secretary, Pacific Gas & Electric Co., 245 Market Street, San Francisco 6, Cal.

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TECHNICAL PAPERS SCHEDULED FOR MAY PUBLICATION

Effect of Sigma Phase vs Chromium Carbides on the Intergranular Corrosion of Type 316 and 316L Stainless Steel, Part 2—Laboratory Investigation, by Donald Warren

A Photo-Electric Information Selector, by R. N. Ride and P. J. Knuckey

Reprint—Classiffication of Topics Relating to Corrosion, by Miss D. M. Brasher

Influence of Temperature on Corrosion Fatigue, by I. Cornet and Simcha Golan

Corrosion Problems in H. F. Alkylation Units—A Report of NACE Task Group, T-5A-6 on H. F. Corrosion

Selecting Corrosion and Scale Inhibitors for Cooling Water, by Hobart Shields, L. V. Sorg and R. L. Stutz

Corrosion Prevention in Tankers and Storage Tanks by Fogging or Flotation With an Inhibitor Solution, by J. C. D. Oosterhout, M. E. Stanley and W. S. Quimby

Corrosion in Sour Water Strippers, by J. F. Mason, Jr., and C. M. Schillmoller

Acetylenic Alcohol-Inhibited Pickling Bath as a Pretreatment Prior to Lining Steel Pipe, by K. N. Edwards, L. J. Nowacki and E. R. Mueller

Erosion of Materials by Cavitation Attack by D. Peckner

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(b) To provide forums and media through which experiences with corrosion and its prevention may be reported, discussed and published for the common good.

(c) To encourage special study and research to determine the fundamental causes of corrosion, and to develop new or improved techniques for its prevention.

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Topic of the Month

Automotive Corrosion Resistance — Past and Present

By LEONARD C. ROWE*

Abstract

The corrosion resistance of automobiles has increased in the past 30 years despite their exposure to generally more corrosive environments. Factors responsible for this longer life include the development of more durable materials, improved machining and surface finishing, and changes in over-all design. 8.9.2

Introduction

A DIRECT COMPARISON of corrosion resistance between the automobile of yesteryear and that of today is obviously impossible. Statistics tend to support the theory that the modern automobile has more built-in protection than its old-time counterpart. In 1925 the life of the automobile was 6½ years and had a total mileage of 25,740 miles.[1] In 1950 these figures were increased to 12¼ years and 103,000 miles. Corrosion resistance was definitely a supporting factor in obtaining this increased service.

Although statistics attest to the durability of cars produced in later years, it is more significant when one considers that this durability has been attained in spite of a gradual intensifying of corrosive conditions. Increased industrial manufacturing has lead to a large output of contaminants into the atmosphere. This in combination with other materials has produced an environment which is more corrosive in nature.

There has been an increase of 135 percent in registered vehicles since 1930. These vehicles accounted for an increase of 163 percent in total vehicle miles of travel. These vast gains can only imply that there has been a change in driver habits and road conditions. One immediate example is the increased use of salt on city streets in the winter. The driver behavior has resulted in a smaller percentage of cars being maintained in garages or shelters, more high speed travel and exposure to variable conditions, and more short trips of the type in which the most corrosive residues are produced.

One can only conclude that conditions are much more corrosive than they were formerly. It can be presumed that the good old car of the past era would fare rather poorly under present conditions.

The earlier car represented a much simpler system to protect than the modern car with its advanced styling, power, and multiple accessories. And yet, in the past it was not unusual to observe devastating types of corrosion which resulted in structural failure, while the modern car shows primarily an appearance failure when corrosion occurs.

Improved Corrosion Resistance

Car structure as a whole has been improved through the use of more durable materials. Part of this advance is due to the development of new materials, and part to improved machining and surface finishing. Metallurgical research has been responsible for improved techniques in casting and metal working. The overall design of the automobile has been improved to give added strength to the chassis and body. If corrosion should occur, there is less immediate effect on any one component.

Corrosion of automotive trim and plated parts has resulted in some criticism in later years. Actually, the total plated film is equivalent or greater in thickness than that previously used. Failures which show up are due to more corrosive conditions, difficulty in obtaining a uniform coating due to design, quantity production, and lack of maintenance by the owner. Brighter things are in store due to the vast increase in technical knowledge. Through the use of new control tests, production standards should be better. Preliminary tests indicate that thicker chromium films give better corrosion resistance. Many parts on the 1959 models have this type of plating. If this arrangement measures up to expectations, it may become standard on all parts.

Automotive body steels are available which offer increased corrosion resistance. In addition, the metal pretreatment coatings have been gradually improved and provide protection to the base metal if the paint should be broken. The new lacquers and enamels not only offer better protection but exhibit less chalking and loss of luster. Clears are now being used to protect the trim molding at the paint interface. This formerly resulted in rust rundown from attack of the trim at the crevice. Zinc-rich paints have reduced the corrosion in the rocker panel area. This is the area where unsightly holes were produced below the door, due to corrosion from the inside out.

Crevices have always presented a corrosion problem. Many of these crevices have been eliminated by combining body or sheet metal sections into single units, thus removing the seam. Drainage and vent holes have been added to keep areas dry longer and to prevent the accumulation of moisture and dirt.

Many cars are beginning to use an aluminum coated steel for muffler and tail pipes. This should give added life to these components. The cooling system has received better attention through customer education and the use of better inhibitors. Engine wear has been reduced through technical improvements and the use of oils with proper additives.

Many advances have been made by the manufacturer in corrosion protection, but a good share of the responsibility remains with the customer. By proper maintenance and frequent washings to remove corrosive substances, the car owner can increase the effectiveness of the preventive measures taken by the manufacturer.

Reference

1. *Automobile Facts.* 9, 7 (1950) Jan.

* Research Laboratories, General Motors Corporation, Detroit, Michigan.

Corrosion Problems Associated With Uranium Refining*

By F. H. MEYER, JR.,* F. J. PODLIPEC* and T. R. KATO*

Abstract

A summary is made of some of the special corrosion problems associated with the production of uranium metal from ore concentrates. After a description is given of processing conditions and existing materials of construction, corrosion rates and effects are related to: (a) variable ore composition, (b) alternate materials of construction, (c) variable operating conditions, and (d) maintenance of product purity. Changes made in the processing system and materials of construction necessitated by the severity of corrosion are discussed. The basic refining process is described and the performance of presently used and alternate materials of construction are compared. 8.10.2

Introduction

THE NATIONAL Lead Company of Ohio operates a plant near Cincinnati which processes uranium ore concentrates received from many parts of the world. Corrosion problems connected with the processing of ore concentrates are complicated by the fact that these concentrates have considerable variation in chemical composition. For example, ores from the Belgian Congo and Australian Rum Jungle are high in chloride. Other ores (those from South Africa, for example) are high in sulfate. All concentrates that have not been previously processed are carefully analyzed for all elements prior to blending and digestion. Other more established concentrates are checked for uranium, fluoride, and chloride content before digestion.

The Ore Refinery

Figure 1 shows the basic steps of the ore refinery process. In the first phase of the operation, the concentrate is digested with nitric acid in tanks equipped with turbine agitators and heating coils. After these tanks are charged with concentrated nitric acid, the ore concentrate is added. Water vapor, nitric acid, and oxides of nitrogen which are evolved are conveyed to the nitric acid recovery area.

The digestion tanks are made of Type 304 L stainless steel, as is the piping leading to and from the tanks. All welds are made with Type 347 stainless steel rod, which has given satisfactory performance. Production tests show a very low corrosion rate, approximately 13 mils penetration per year.

The valves and pumps used in this stage of processing are constructed of Durimet 20 or Duriron. Erosion caused by the ore slurries is a problem, and pumps and control valves must be replaced frequently.

Fluorocarbons are used as gasketing materials for this hot nitric acid service. Performance has been exceptional insofar as corrosion is concerned.

In phase two of the ore refinery process, the uranium is recovered directly from the digestion slurry by solvent extraction in perforated-plate pulse columns. The organic extractant, tributyl phosphate in an inert purified kerosene diluent, extracts uranium from the aqueous slurry in the primary extraction column.

Both vertical-piston-type and fluorocarbon-bellows-type pulse generators are used in the pulse columns. Corrosion in the piston-type pulse unit has occurred, due, apparently, to galvanic action between the graphite piston rings and the Type 304 cylinder wall and piston. The bellows-type pulse unit has not contributed to the corrosion problem. The bellows are made of polytetrafluoroethylene and were operated for about 6 million cycles before rupturing. Failure after that number of cycles was attributed to design.

The uranium-bearing extract from the primary extraction column is pumped to a scrub column, where it is contacted with deionized water to remove metallic impurities and nitric acid. During the scrubbing operation, essentially all of the residual acid and metallic impurities, together with small quantities of uranium, are transferred from the organic phase to the aqueous phase. The scrubbed organic stream then cascades through a re-extraction column where an aqueous stream re-extracts the uranium, producing an aqueous uranyl nitrate product and a stripped uranium-free organic stream. No significant corrosion problems have been observed in this phase. The pulse columns are made of Type 304 L stainless steel.

Concentration of Aqueous Uranyl Nitrate

The third phase of the refinery process consists of the concentration of the aqueous uranyl nitrate. Initial evaporation is carried out in tubular evaporators; subsequent evaporation to 100 percent uranium nitrate hexahydrate takes place in boildown tanks. Tanks in this phase are constructed of 304 L stainless steel with Type 347 stainless steel steam coils. A special welding technique has been developed which has greatly minimized end grain corrosion and erosion.

Table 1 shows the rates of corrosion obtained in the boildown area. Samples of the 300 series stainless steels were exposed for four months under operating

TABLE 1—Corrosion Rates of Stainless Steels*

Stainless Steel Type	Corrosion Rate (Mils Per Year Penetration)
AISI 347	0.56
AISI 304	0.36
AISI 304 L	0.36
AISI 316	0.33
AISI 309	0.35

* Condition: 4 months' exposure in an operating boildown tank.

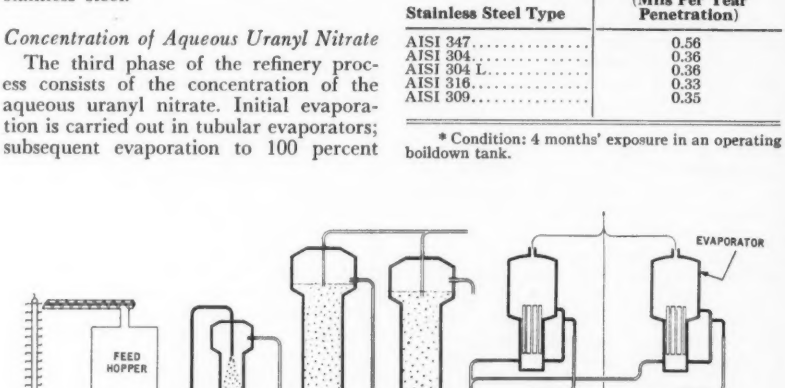

Figure 1—Uranium ore refinery process.

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* National Lead Company of Ohio, Cincinnati, Ohio.

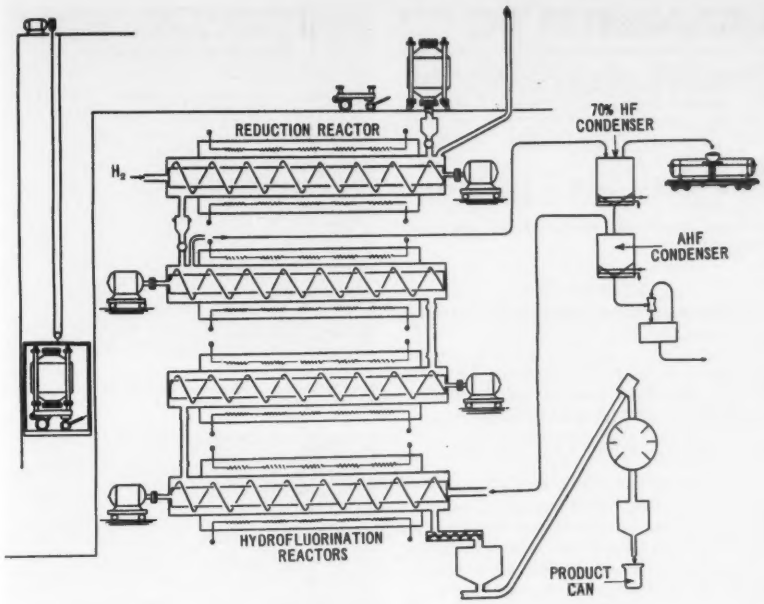

Figure 2—Green salt process.

conditions. As the table indicates, there is no large difference in the corrosion rates of the 300 series steels. Because of its low cost and ease of fabrication, Type 304 L stainless steel is used in the National Lead Plant in new installations.

Some contamination from iron and nickel was originally encountered in boiling down the uranyl nitrate because of exposure of the steam coils to uranyl nitrate vapors. This occurred whenever steam was passed through the coils with the tank not completely full. The procedure has been altered so that the coils are covered with uranyl nitrate prior to heating. Since this was done contamination from this source has ceased.

Denitration Process

The fourth phase of the refinery process is the denitration of the fused hydrated uranyl nitrate to form uranium trioxide. This is accomplished by heating the salt in gas-fired pots. Type 347 stainless steel has been used satisfactorily for this operation. Difficulties such as pot failure at the knuckle radius and bottom center have been encountered as a result of cracking of the pots, but they were attributed to thermal cycling and overheating in the batch-type operation. Thermocouples are now being installed on pot bottoms in place of in-flue gas for better temperature control. Programmers also have been installed for improved firing and cooling control.

Nitric Acid Recovery

In the fifth phase of the refinery process, the nitric acid is recovered from the primary extraction column slurry (which contains free acid and various nitrate compounds). This material is concentrated in evaporator units to a viscous material and then is spray calcined to recover additional nitric acid by the absorption of NO_2 gases in an absorber system. The evaporator overhead nitric acid vapor is concentrated in a 12-plate vacuum distillation unit. The Type 304 and Type 309 stainless steels used in calcination have corroded. This can be attributed to the effects of moist NO_2 fumes. A drum drier having a chromium-plated drum has been constructed and to date has given satisfactory concentration of the raffinate.

The combined NO_2 vapor fumes from the digestion area, denitration area, and other operations are sent through the absorber system of the nitric acid recovery system. The recovered acid is concentrated for reuse in the digestion area.

Severe equipment corrosion has been encountered in the nitric acid recovery system because of small quantities of chlorides and fluorides carried by the vapor streams. Chloride removal by oxidation is accomplished by the use of Welsbach ozone generators. Chloride-bearing liquor is extracted from the nitric acid concentrator at a point where the nitric acid concentration approaches 20 percent and chloride levels are highest. This liquor is passed through an ozone sparge unit which oxidizes about 75 percent of the chloride to chlorine. The acid is then returned to the concentrator as internal reflux.

Effect of Fluorides

Greater corrosion of the concentrator is caused by high fluoride levels than by chlorides. Table 2 shows the effect of high fluoride content (1000 ppm) on test strips placed in the nitric acid concentrator during a six-week period of operation. Note the high corrosion rate of

TABLE 2—Corrosion Rates of Test Specimens Exposed in a Nitric Acid Concentrator for a Six-Week Period[1]

Material	Corrosion Rate (Mils Per Year Penetration)	
	Fluoride Level[2] (Over 1000 ppm)	Fluoride Level[3] (Under 200 ppm)
Durimet 20	64	55
Durichlor	22	0.4
Duriron	26	0.4
Stainless Steel 304	212	30.0
La Bourr 55	2300	*
Elkomet K	2385	*
Inco 803	78	*
Tantalum	*	0.01
Stainless Steel 309 Cb	*	25.0
Stainless Steel 309 SCb	*	25.0
Stainless Steel 347	212	45
Stainless Steel 321	*	65
Hastelloy C	*	Failed
Chlorimet 3	*	Failed

[1] Nitric acid concentration: approximately 60 percent; temperature: 250F.
[2] No Complexing.
[3] With Complexing.
* Not Determined.

Type 304 stainless at this fluoride level. Because of the lack of a suitable material from which tanks could be constructed, the fluoride ion was complexed by the addition of Al_2O_3 to slurry in the ore digestion tanks. This formed an AlF_3 or AlF_6^{3-} complex which had a much lower volatility in acid solutions than did the free fluoride ion. The ratio of five parts of aluminum (as Al_2O_3) per part of fluoride has been found sufficient to suppress the fluoride volatility in the evaporation step. This procedure removed about 90 percent of the fluorides introduced to the digestion process.

The effect of this addition is shown in the second column of Table 2, where the fluoride level was less than 200 ppm for a six-week period. From these results and on the basis of costs and ease of fabrication, it is evident that stainless steel Type 304 L is best. Note also that Durichlor and Duriron perform most satisfactorily. They are recommended as materials of construction for pumps and valves to be used in this service.

To further decrease corrosion in the concentrators, new units will operate under a vacuum of 200 mm. Laboratory tests have indicated that this will appreciably reduce corrosion rates.

The Green Salt Plant

Figure 2 shows the basic steps of the green salt process. Orange oxide (UO_3, uranium trioxide) from the ore refinery is fed into a reduction reactor containing a screw conveyor and is there reduced with hydrogen formed from dissociated ammonia. The chemical reaction in the reduction reactor is as follows:

$$UO_3 + H_2 \rightarrow UO_2 + H_2O$$

The original reactor tubes were fabricated from Type 309 Cb stainless steel and the shafts were fabricated from Inconel. A process change was made early in plant operations and the nickel content of the green salt (UF_4) increased immediately. Investigation revealed that the Inconel shaft had corroded. Later laboratory tests indicated that hydrogen

TABLE 3—Corrosion Rates of Various Alloys Exposed to the Reduction Reactor During Processing of Sulfated Orange Oxide

Material	Corrosion Rate, MDD (Gain)			Corrosion Rate, MPY (Gain)		
	Rack 1	Rack 2	Rack 3	Rack 1	Rack 2	Rack 3
Inconel	—3.24	—183.78	—0.776	—0.55	—31.2	+0.13
Hastelloy C	+0.29	Failed	+0.29	+0.05	Failed	+0.05
Illium R	—0.44	.....	+0.09	—0.07	.....	+0.02
Stainless Steel 304	+5.18	— 5.47	+4.08	+0.94	+ 0.99	+0.74
Stainless Steel 316	—2.2	— 2.8	+2.4	—0.40	— 0.51	+0.44
Stainless Steel 309 Cb	+0.81	+ 4.8	+1.0	+0.15	+ 0.87	—0.18

Average Reactor Temperature (Excluding Reaction Zone): 1200F.
Exposure Time: 46 Days
Rack Positions: 1—One foot from feed end.
2—Middle of reactor (reaction zone).
3—One foot from discharge end.
Average Process "Atmosphere": 1 mol H_2 to 1 mol N_2 to 2 mols water vapor; variable sulfate and sulfide content.

sulfide was probably formed in the reaction zone, severely attacking the Inconel shafts and causing the increased nickel content of the green salt.

Table 3 shows the corrosion rates of various alloys exposed to the reduction reactor environment while sulfated orange oxide was being processed. Note the high corrosion rates of the high-nickel alloys. As a result of these tests, stainless steel Type 316 is now used for the shaft and ribbon in this reactor, and Type 309 (columbium stabilized stainless steel) is used for the shell. Performance to date has been satisfactory and impurity contamination has been minimized.

The brown oxide (UO_2) formed in the reduction reaction is hydrofluorinated with anhydrous acid at progressively increasing temperatures in a series of three hydrofluorination reactors containing screw conveyors. There is a modified ribbon-flight type screw running through the center of each reactor tube. The exothermic chemical reaction in the hydrofluorination reactors is as follows:

$$UO_2 + 4\,HF \rightarrow UF_4 + 2\,H_2O$$

Table 4 shows the corrosion rates of Inconel in the hydrofluorination reactor. These reactors are essentially the same as the previous reactors, but are constructed of Inconel, with either Illium or Hastelloy C reactor screws. The system must be capable of withstanding high temperatures in an HF atmosphere. Inconel shows a corrosion rate which is very satisfactory. The performance of the Hastelloy C and Illium ribbon supports also has been satisfactory.

In the control of corrosion, continued vigilance is required due to changes in the composition of ore concentrates and changes made in the process in order to increase the productivity.

TABLE 4—Corrosion Rates of Inconel Exposed in a UF_4 Reactor for a Period of 80 Days of Plant Operation

Specimen Location*	Penetration (MPY)
Feed End	0.005 0.025
Intermediate	0.097 0.120
Intermediate	0.189 0.190
Intermediate	0.166 0.167
Intermediate	0.249 0.276
Discharge End	0.277 0.300

* The specimens were equally spaced over the reactor length.

DISCUSSION

Question by M. A. Scheil, A. O. Smith Corporation, Milwaukee, Wisconsin:

Mr. Meyer has indicated corrosion rates of a large number of materials in the concentration of uranium ores. I would be particularly interested in whether any use has been made of tantalum in the author's process since it is noted in the paper that tantalum has a very low corrosion rate.

Reply by F. H. Meyer, Jr.:

We have not used tantalum as a material of construction due to the economic factor. All our equipment is large scale chemical processing equipment and the use there of tantalum would not be economically feasible as compared with stainless steels.

Any discussion of this article not published above will appear in the December, 1959 issue

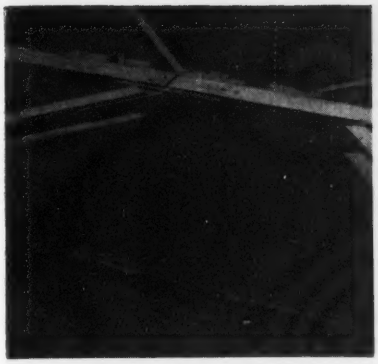

Figure 1—Hot spray vinyl coating on waste water separator.

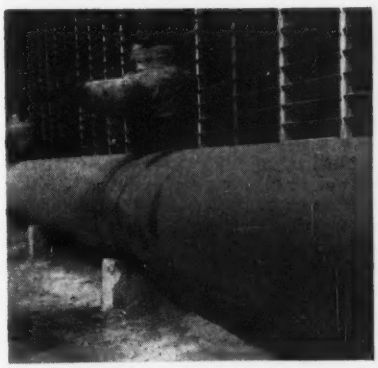

Figure 2—Hot spray vinyl coating on large water lines adjacent to a cooling tower.

Figure 3—Application of catalytic cured phenolic coating to inside of an exchanger channel.

Oil Refinery Applications Of Thick-Film Synthetic Coatings*

By R. W. MAIER, W. B. COOK and R. B. MacQUEEN

Abstract

Three recent developments in synthetic protective coatings have made possible relatively thick film applications per coat without resulting in sags, runs or skin-dry wrinkling. These developments are hot spray application, catalyst-cured coatings and high-film-build alkyd paints.

The following are discussed: hot spray vinyl, catalyst-cured epoxy, catalyst-cured phenolic, catalyst-cured epoxy-modified phenolic and high-film-build alkyd coatings and also the advantages derived from their application to oil refinery equipment. Coating problems involved in protecting oil refinery equipment are noted, and field experiences with thick-film applications on various equipment surfaces exposed to a wide range of contact media and conditions are described.

With the exception of high-film-build alkyd paint, field data are given in tables and are described in detail in the text. 5.4.5

Introduction

UNTIL SEVERAL years ago, the terms "thick coatings" or "mastics" referred to black coal tar or asphaltic materials. These materials have done excellent jobs of protecting certain surfaces in specific exposures when properly applied. For many years, thick-film coal tar coatings have been recognized as materials which would provide excellent protection to pipe lines, and the asphalt mastics have been proven to possess good weather-sealing properties for use as coatings over insulation and to afford long-term protection to steel surfaces such as structural members in mild acid and high moisture areas. However, these natural coatings, as all coatings do, have limited resistance to chemicals, solvents, temperature, etc. Synthetic coatings on the market today can be applied in relatively thick films with wider range of resistance to contact media than the coal tars and asphalts.

Developed during the past 25 years, many of these synthetic or plastic resins in tests have proved to be more resistant to many corrosive conditions than materials previously known. When first formulated into coatings and for years afterward, some resins had the shortcoming of producing only thin films and often required baking of each coat for curing.

In oil refinery applications such as lining and coating of containers, package decorating and coating of caps and closures, these shortcomings were of little consequence because coatings were applied indoors under controlled production line techniques. A film thickness of one mil (.001 inch) or less applied in one coat was sufficient to resist the conditions to be encountered. Field conditions, however, presented different problems and made conformity necessary with the universally discussed minimum of 5 mils film thickness or at least a 3-coat application for industrial plant painting or maintenance coating work. To obtain 5 mils with the synthetics, usually more than 3 coats were required; for vessel lining work which needed a film thickness of 5 mils or more, five or more coats were often necessary.

Oil refineries operate continuously around the clock. Equipment is taken out of service piecemeal after relatively long operating periods and, because of economics involved, is held out of service only long enough for necessary cleaning, repairs and protective coating work that is impractical while the equipment is in service. Therefore, measures which allow application of required film thickness of adequately resistant coatings in the least number of coats are advantageous.

Recent developments in coatings make it possible to apply synthetics in multi-mil film thickness per coat. Three of the most outstanding advancements have been development of hot spray application, advent of catalyst-cured coatings and high-film-build alkyd formulations. In addition to the economic advantage of quickly returning equipment to production service, these developments also reduce labor costs by requiring fewer coats.

Refinery Applications

These developments have been used in coating operations at Gulf Oil Corpora-

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TABLE 1—Philadelphia Refinery Application of Hot Spray Vinyl Coatings

Application	Environment	Surface Preparation	Method of Application	Date of Application	Number of Coats	Total Film Thickness (Mils)	Comments and Results
Waste water separator	Water, oil heat	Sandblast	Cold spray primer hot spray finish	1955	1 Primer 1 Finish	10–12	Excellent condition after 1½ years
Exterior of 42 inch water line adjacent to cooling tower, 6 inches above ground	Water spray with water treating chemicals	Sandblast	Cold spray primer hot spray finish	1956	1 Primer 1 Finish	8	Looks promising and may be best answer to this problem

R. W. MAIER is corrosion engineer with Gulf's Manufacturing Department in Pittsburgh. Formerly he did research and sales work in protective coatings. He has a BS and MS in chemical engineering from the University of Pittsburgh and is a member of NACE.

W. B. COOK is affiliated with Gulf Oil Corporation's Port Arthur refinery in coatings work. He is a graduate of Texas A & M College and is a member of NACE.

R. B. MacQUEEN is foreman of the Builder's Department at Gulf Oil Corporation's Philadelphia refinery. He is responsible for the application of paints and protective coatings to all refinery equipment.

tion refineries, and the results have been encouraging. Pertinent data regarding some of these applications are shown in Tables 1, 2 and 3.

Table 1 shows refinery field test applications of hot spray vinyls.

Table 2 presents data on typical applications of a catalyst-cured phenolic coating and a heavy consistency catalyst-cured epoxy coating.

Table 3 covers a number of catalyst-cured epoxy-modified phenolic applications.

Applications of a fourth type, high-film-build alkyds, were similar; data on them are not presented in tabular form.

A fifth type, a thick-film coal tar-epoxy coating, recently developed is being field tested at Gulf's Philadelphia and Port Arthur refineries. This material was applied successfully and shows promise of providing good protection to surfaces in certain exposures, but insufficient test data are available at present.

Experienced specific applications with the first four of these classifications of thick film coatings are reported below.

Hot Spray Vinyls

Hot spray vinyls differ from conventional vinyls in that they have higher percentage of solids or non-volatiles and lower solvent content. In cold spray application of conventional vinyls, the coating as received must be cut with a high volume percentage of relatively expensive special thinner to eliminate cob-webbing and deposit of dry overspray. This cutting results in the deposit of relatively thin films. Fast evaporation of thinner and solvent from the wet film is likely to result in pin-holes in the dry film. Application of multiple coats, however, should remedy this deficiency because pin-holes probably will not occur at the same points in different coats. In hot spray application, the uncut coating is heated to approximately 160 F by a specially designed heater ahead of the spray gun. This reduces the viscosity, allows for proper atomization and causes immediate evaporation of much of the solvent, thus reducing pin-holing and allowing a thick, tight film to be deposited with little or no sag. Applications are shown in Table 1.

Waste Water Separators

Metal surfaces at the waste water separator are exposed to water, waste oil and temperature variations. In the past, many types of coatings—from oil-based paints and conventional vinyls to heavy bitumens—have been tried on these surfaces. All these coatings failed within a few months, due either to direct attack on the coatings or to insufficient film thickness. The hot spray vinyl, applied to these surfaces as shown in Table 1 was in excellent condition after more than a year of exposure. Figure 1 illustrates this application.

Cooling Tower Piping

Water circulation pipe lines adjacent to cooling towers in refineries for transferring warm water from condensers and coolers at the processing units to the cooling tower are exposed to a warm, humid atmosphere from the spray of treated water. Although a system of sandblasting, priming with an oil type inhibitive primer and application of asphalt mastic to at least ⅛-inch thickness dry film, all accomplished in a dry atmosphere, have protected these lines for several years, a thick-film hot spray vinyl application might provide longer-lasting protection. Such an application was made at the Philadelphia refinery and reported in Table 1. Figure 2 illustrates this application.

Catalyst-Cured Phenolics and Catalyst-Cured Epoxies

For many years, phenolic coatings have been considered excellent for resistance to moisture penetration, acid attack and elevated temperatures but were usually associated with the terms "thin films" and "baking." A new development in phenolic coatings was introduced that made curing possible by addition of a liquid catalyst as a substitute for baking and could be applied by multiple-pass to a film thickness of 12 mils per coat without resulting in runs or sags. At about the same time, epoxy resin was coming into prominence, and catalyst-cured epoxy coatings were being developed. Some of these coatings contained no volatile matter, being made up to 100 percent film-forming material.

Exchanger Lining

During a still shutdown at the Philadelphia refinery in October, 1953, a channel of one of the exchangers was sandblasted and given three brush coats of a catalyst-cured phenolic coating at a film thickness of about 4 mils per coat. Drying was relatively slow for this type of coating, and the final coat was not completely set by the time this channel was installed. However, when this still was shut down again in November, 1954, the coating was found to have developed a hard finish and to be in excellent condition after a year's service, showing no sign of attack from the cooling water. This application is given in Table 2.

Certain neoprene-based and bitumen-type coatings had been tried previously in this service. Although they afforded some protection, they were not as successful as the catalyst-cured phenolic. Since this initial test, many more exchanger channels and heads have been coated with the phenolic material, using two coats to

TABLE 2—Philadelphia Refinery Applications of Catalyst-Cured Phenolic and Catalyst-Cured Epoxy Coatings

Application	Environment	Surface Preparation	Method of Application	Date of Application	Number of Coats	Total Film Thickness (Mils)	Comments and Results
Interior of exchanger channels	Warm cooling tower water	Sandblast	Brush	10/53	3	12	Catalyst-cured phenolic. Excellent condition after one year's service. Decided to try two coats (see next Item).
Interior of exchanger channels	Warm cooling tower water	Sandblast	Brush	1954	2	8–9	Catalyst-cured phenolic. A number of applications have been made and it is now established that at least 2 years excellent protection is afforded.
Exterior seam of old riveted tank to stop seam "weepage"	Foamite	Sandblast	Brush and trowel	8/56	See Text	See Text	Catalyst-cured epoxy. No further "weepage."

obtain 8 to 9-mil film thicknesses. Figure 3 shows a typical application. Experience indicates this type of application will protect these surfaces for at least two years.

Tank Seam Sealer

Catalyst-cured expoxies have been formulated at heavy consistencies as tank seam and rivet caulking compounds. At the Philadelphia refinery, as shown in Table 2, a relatively old, riveted foamite storage tank which had begun to "weep" at some of the seams was drained, and seams were sandblasted and sealed with a thick brush-and-trowel application on the outside with epoxy caulking compounds in August, 1956. To date the seams have held, and no further "weeping" is in evidence.

Catalyst-Cured Epoxy-Modified Phenolics

Epoxy-modified phenolics of the catalyst-cure type were developed along with the catalyst-cured phenolics and the epoxies.

These coatings are formulated into different grades in which the ratio of epoxy and phenolic resins vary. Generally, the manufacturers' recommendations have been followed as to which grade should be used for specific exposures. When one manufacturer's recommendations for these coatings were studied, the claim was significant that as coatings for oil refinery applications, unlike the straight phenolics, they had good resistance to alkali in addition to resistance to acid attack, and, unlike the vinyls, good resistance to temperatures above 150 F immersed and far above 180 F dry. These properties, plus thick-film atmospheric temperature application catalyst-cure characteristics, made the coatings usable for protecting refinery equipment exposed to corrosive conditions.

Extensive field testing of this modified type coating has been conducted at the Port Arthur refinery since December, 1952, when the first multi-pass spray application was made in one coat as an interior lining in a horizontal (10-foot diameter by 70-foot length) moisture-bearing-propane storage drum at the propane deasphalting unit. Pertinent data regarding these applications are shown in Table 3.

Propane Drum Lining

The propane drum at the deasphalting unit was built in 1944 with a corrosion loss allowance of ⅛ inch. Periodic inspections showed a gradual pitting and metal loss, mostly along the bottom. In April, 1950, pits as deep as 3/32 inch had developed in this area of the surface. At that time, the drum's interior was sandblasted, and a multiple-coat baking type phenolic lining was spray applied and baked. An inspection made after this drum had been in service for nine months showed at least 90 percent of the lining to be intact, but the coating was blistered badly and peeled in a strip about three feet wide along the bottom of the tank. This failure was believed to be due to underbaking which often occurs in a large vessel field-baking operation. Since there had been no metal loss, the drum was returned to service. A year later the bottom third was strip lined with plain steel. Sandblasting and recoating the interior were planned for a later time when operations would permit the drum to be out of service.

Sandblasting and re-coating were done in December, 1952. The coating used was a cold setting, catalyst-cured, epoxy-modified phenolic applied by multi-pass cross-spray in one coat to a film thickness that varied from 4 to 12 mils. After 9½ months' service the coating was in good condition except for small, scattered blisters in about 50-square-foot area near the propane inlet on one side wall. The coating was brittle at these blisters. Failure in this area was attributed to low temperature shock and erosion from the incoming propane under pressure.

This blistered area of the coating was repaired by sandblasting and brush re-coating with one prime and two heavy finish coats of the same epoxy-modified phenolic coating. Several 1 foot x 3-foot pieces of reinforcing glass cloth fabric were embedded between the two finish coats. This coating was inspected again in January, 1956, after two years and three months of service since the last repair work and three years since the base cold setting modified epoxy coating was applied. About 50 percent of the coating had peeled from the surface, but the only serious metal loss was in a few pitted spots in the bottom strip lining. These pits were filled with a cement mortar before the drum was returned to service for another two-year period. The portion of the coating where the glass cloth was embedded was completely intact.

Lining For Six New Propane Drums

Results of this drum lining application were sufficiently encouraging to justify more applications. A thicker film was planned for better protection. Therefore, when six new 10-foot diameter by 100-foot length propane drums were built with practically no corrosion loss allowance early in 1955, the interior of each drum was sandblasted to a white metal finish and an anchor pattern pit depth of 2 mils and lined with one 6-mil multi-pass spray coat and one 9-mil coat of a more elastic formulation of catalyst-cured epoxy-modified phenolic coating.

One of these drums was taken out of service and inspected after two years' service. There were no visible breaks in the coating, but about 15 percent of the coating surface was puffed out in a pattern of blisters scattered over the entire drum surface. The majority of these blisters ranged in size from ½ to 2½ inches in diameter, with only a few as large as 4 to 5 inches. Two of the largest blisters were broken to determine the condition underneath the coating. Both coats had been lifted and the coating was rather brittle, but there was no apparent moisture underneath. The metal had the appearance of new steel, freshly sandblasted to a gray commercial grade finish.

The coating at the smaller blisters was flexible and could be pressed back to the steel surface without rupture by thumb pressure. There had been some slight penetration through pin-holing in the coating. Release of service pressure when the tank was emptied and the subsequent build-up of gas pressure underneath the coating at the spots of penetration probably caused the blistering.

This drum was returned to service without any coating repair work and scheduled for another interior inspection after two or three more years of service because the inspectors thought service pressure would force the unbroken blistered coating back against the steel. The spots at the two broken blisters will be checked periodically from the exterior for metal loss with the Audigage.

From knowledge gained from the propane drum lining, field testing of catalyst-cured epoxy-modified phenolic coating at the Port Arthur refinery was extended to applications on other equipment.

Caustic Resistance

That phenolic type coatings are not resistant to caustic soda is recognized. Surprisingly, one manufacturer recommended an epoxy-modified phenolic for this service. However, on some panel tests, this phenolic was found to have resistance. Field tests were made as a caustic tank interior seam lining and as an exterior coating on a waste oil treating tank subject to 200 F heat and condensed caustic-bearing oil vapor rundown. In 1954, some of the seams of an old 40-foot diameter, 25-foot height, riveted steel tank in 45-degree Bé caustic soda solution service began to leak slightly. After the tank was emptied and cleaned, interior of the seams and rivet heads were sandblasted and brush primed with the catalyzed epoxy-modified phenolic. The angles at the overlap of steel plates were beaded by caulking gun application with a catalyzed epoxy-modified phenolic caulking compound followed by brush-and-spray application of two coats of the catalyzed coating with a layer of reinforcing glass mesh embedded between each coat. No leakage at the seams and rivets of this tank has occurred for more than four years after the coating was applied.

Inspection of the interior, made after three years of service, showed the coating to be intact except for slight curling at the edges several inches from the seams. Durability of the application made to the exterior of the waste oil treating tank continues to be excellent after more than four years with no indication of attack by the hot caustic-bearing condensed oil vapor rundown. However, the approximately 200 F temperature and exposure to the sun has faded the black color to a uniform blue-black.

Acid Resistance

Field tests have proved this type of coating to be resistant to sulfuric acid solutions including those at elevated temperatures, but applications on surfaces exposed to hydrochloric acid attack have been considered failures. As shown in Table 3, applications made to the exterior of a lead-lined, open-top tank used for open steam cooking of sludge from sulfuric acid treatment of lubricating oils and also made to

TABLE 3—Port Arthur Refinery Applications of Catalyst-Cured Epoxy-Modified Phenolic Coatings[b]

Application	Environment	Surface Preparation	Method of Application	Date of Application	Number of Coats	Total Film Thickness (Mils)	Comments and Results
Interior of horizontal propane storage drums (1 drum 10′ Dia. x 70′ long) (6 drums 10′ Dia. x 100′ long)	Moisture-bearing propane under pressure	Sandblast to white metal	Multi-pass spray	 12/52 4/55	 1 2	 4–12 14–15	 See text. See text.
Inside seams of old riveted caustic tank to stop "weepage"	45° Bé. NaOH solution	Sandblast to white metal	Brush, spray & caulking gun	3/54	See text	See text	No "weepage" had occurred at the coated seams for over 4 years. Interior inspection 5/57 showed coating to be intact.
Exterior of waste oil treating tank	Spillage and fumes from caustic treatment at 200°F.	Sandblast to white metal	Multi-pass spray	4/54	2	8–10	Durability excellent for over 4 years but heat and sunlight has faded black color to blue-black.
Exterior of lead-lined open-top sulfuric acid sludge "cooking" tank	Spillover of hot sulfuric acid-bearing heavy oil sludge	Sandblast to white metal	Brush and spray	7/54	2[a]	8–10[a]	About 70% of the coating deteriorated (carbonized) after affording excellent protaction and good appearance for 3 years.
Interior of two old "Everdur" acidic water settling drums which had lost their corrosion allowance	Acidic (sulfuric) water dropout from light oil distillate	Sandblast to bright metal	Brush and spray	4/55	2	8–10	Looked good when inspected 2/57, with only a few scattered small breaks in the upper half. Lining was reinforced with another coat after minor hand tool cleaning.
Underside of roof of small clay slurry tank at Clay Contact Plant	Exposure to hydrochloric acid-bearing oil vapors	Sandblast to white metal	Multi-pass spray	12/55	3	10–12	Had failed on inspection after a 6-month run of the unit. A polyester glass fiber application made afterward to new roof has afforded satisfactory protection.
Underside of roof of hydrochloric acid-bearing distillate tank at Clay Contact Plant	Exposure to hydrochloric acid-bearing oil vapors	Sandblast to white metal	Multi-pass spray	12/55	3	10–12	Had failed on inspection 12/56.
Interior of open top demineralized water storage tank (50′ Dia. x 30′ high)	Atmospheric temperature demineralized water	Sandblast to white metal	Brush and spray	6/55	3	9–11	Excellent after three years of service.
Interior of open top steam condensate tank (60′ Dia. x 30′ high)	Causticized steam condensate at temperature up to 180°F.	Pickle and sandblast to white metal	Brush and spray	9/54	3	9–11	Became overcoated in service with somewhat oily, caustic film which affords some protection. However, coating appears to be in good condition after 4 years, 3½ of which has been steam condensate service.
Interior of new steel horizontal drinking water tank (10′ Dia. x 25′ long)	Potable water	Sandblast to white metal	Multi-pass spray	12/55	3	9–11	Was slow to cure in 40-50°F. temperature, and this was indicated by bad taste and odor. Coating was then forcecured by heating the water with open steam at 170-190°F. for several days until no taste and odor was detectable after draining and refilling. No inspection of the condition of coating has been made.
Interior of 2 elevated open top fire protection water tanks	Brackish water	Sandblast to white metal	Multi-pass spray	10/56	3	9–11	No inspection made since application.
Interior of corrosion pitted shells and channels of 4 product coolers at Platformer Unit	Warm cooling tower treated water	Wet sandblast	Multi-pass spray	1/57	3	12–15	Inspection during unit turnaround 11/57 showed small, scattered blisters but no appreciable metal loss. Coating was intact at the old pits.
Heads and channels of exchangers for atmospheric-vacuum crude still	Warm cooling tower treated water	Wet sandblast	Multi-pass spray	5/57	3	12–15	Inspection during unit turnaround 5/58 showed small, scattered blisters but no appreciable metal loss.
Several applications to hot oil coils cooled by salt water spray at Clay Contact Plant	Heat and salt water (oil enters coils at approximately 450°F.)	Sandblast to white metal	Multi-pass spray	1955–1958	2	6–8	This satisfactorily protects these coils between six months turnarounds of the process units.
Interior of the following indicated portion of the shell and entire length of roof support columns of cone roof light oil blending tanks as follows:	Alternating light oil-moist air condensation and vaporization. Intermittent rising and falling light oil liquid level accompanied by mechanical mixer agitation at high liquid level.	Sandblast to white metal	Multi-pass spray and caulking gun				
1-120′ Dia. x 48′ high tank, upper half of shell				2/54	2	10–12	Scattered breakdown. Very good protection for more than 4 years. See text.
1-140′ Dia. x 48′ high tank, upper half of shell				2/57	2	10–12	Results unknown. Tank in continuous service.
1-160′ Dia. x 48′ high tank, upper half of shell				2/58	2	10–12	Results unknown. Tank in continuous service.
1-160′ Dia. x 48′ high tank, upper half of shell				5/58	2	10–12	Results unknown. Tank in continuous service.
1-117′ Dia. x 42′ high tank, entire shell				1/55	2	10–12	Results unknown. Tank in continuous service.
Induced draft cooling tower fan assembly. Aluminum fan blades.	High humidity fog atmosphere from chemically treated water	Sandblast to white metal	Spray and brush	10/54	2	6–8	Affording good protection more than 3 years after application.

TABLE 3 (Continued)—Port Arthur Applications of Catalyst-Cured Epoxy-Modified Phenolic Coatings[b]

Application	Environment	Surface Preparation	Method of Application	Date of Application	Number of Coats	Total Film Thickness (Mils)	Comments and Results
Hubs of new, thinly coated fans for forced draft cooling tower	High humidity fog atmosphere from chemically treated water	Hand sand	Spray	1/55	1	4–5	Affording good protection more than 3 years after application.
Interior of 2 transformer oil tank cars	Transformer oil	Rotary wire brush	Multi-pass spray	3/55	1	3–4	Coating in good condition after more than 3 years of service. Coating facilitates cleaning and prevents contamination of this hightly refined product.
Interior of corrosion inhibitor charge pots for process units	Corrosion inhibitors	Wire brush	Brush	1955–1956	2	6-8	Has prevented contamination of inhibitors from rusting of the pots for several years. Pots were fabricated from new steel piping.
Interior unglazed hollow tile walls	Atmosphere inside buildings	Detergent wash	Multi-pass spray	4/55	2	3–4	Clear coating applied. Is unaffected in any way after more than 3 years, Has retarded soil accumulation. Coated unglazed tile costs about ¾ that of glazed and has additional benefit that mortar is glazed.
Top side of process unit concrete deck working area to stop seepage of rainfall into control room below	Normal rainfall. Abrasion and oil spillage during exchanger cleaning during unit turnrounds.	Chemical cleaning and neutralization	Multi-pass spray	1955	3	8–10	Clear, unpigmented, first coat thinned for penetration and sealing of the pores. Next two coats were pigmented, into which fine grain silica sand was sprinkled to make skidproof. Has stopped rainfall seepage for more than three years.

[a] Protruding ends of bolts for securing lead lining were coated with heavy consistancy tank sealing compound.
[b] All surfaces are steel unless otherwise noted.

the interior of enclosed vessels used for settling out of water from "washing" sulfuric acid treated light oils, gave good protection to metal surfaces for several years. These coatings, however, failed when applied to vessel interiors exposed to hydrochloric acid-bearing oil vapors.

Water and Steam Condensate Immersion

Three-coat applications made to the interior of a flat-bottom, open-top demineralized water storage tank and a flat-bottom, open-top steam condensate tank at a total film thickness of 9 to 11 mils have afforded apparently good protection to the steel. Although these tanks have not been emptied completely since applications were made more than three or four years ago, respectively, visual inspections made after this length of service when the water levels were lowered to about half the height of the tanks show the coatings to be intact. However, caustic solution is introduced constantly to the steam condensate tank for pH control, and this forms a protective film on the coating. Figure 4 is a photograph of the interior of the coated demineralized water tank, taken after more than two years of service when the water level was about four feet from the top. Figure 5 shows corrosion scale build-up and spalling of the scale above the water level in an uncoated treated water tank (pH about 5.0) which was built at the same time as the coated tank in Figure 4.

The interior of a new 10-foot diameter by 25-foot length, horizontal drinking water storage tank was standblasted and spray coated with three coats of the catalyzed epoxy-modified phenolic recommended by the manufacturer for potable water service use in December, 1955, during 40 to 50 F atmospheric temperatures. Although the coating had about ten days of curing before being put in service, twice the recommended curing time for 70 F ambient temperature, the water had a bad taste and odor after the tank was filled and drained several times. This condition was due to insufficient curing at low temperature and was remedied by filling the tank, introducing open steam, heating to 170 to 190 F for several days and draining the tank.

In January, 1957, during a shutdown of a platformer unit, the water side interior of the shells of four product coolers or condensers was found to be badly pitted. These were wet standblasted and a 3-coat spray application of catalyzed opoxy-modified phenolic was made to a total film thickness of 12 to 15 mils. These were inspected during the next turnaround in November, 1957. Although some coating was removed from the bottom from sliding the tube bundle in and out and although there were scattered, small unbroken blisters in the coating, no further pitting had occurred. The coating over the old pits was well adhered and affording good protection.

During this turnaround and a previous turnaround of an atmospheric-vacuum crude still, a number of exchanger heads and channels to be exposed to cooling water were wet standblasted and coated similarly. A recent inspection of the heads and channels at the atmospheric-vacuum crude still, made during a shutdown after about a year of service, showed this coating to have afforded good protection. There were some small scattered breaks in the coating where blistering had occurred, but metal loss and pitting were negligible. The breaks were re-blasted and recoated with the same material. Apparently, such surfaces need to be recoated annually with this type coating. Savings from the protection afforded, however, far surpass the cost involved.

Interiors of Light Oil Tanks

Accelerated atmospheric type corrosion of cone roof tank interior shells in light oil service is a general concern to the oil refining industry. Tandy agrees that these tanks may have corrosion rates as high as 20 mpy.[1] At the Port Arthur refinery, corrosion rates on upper rings of light oil blending tanks have been almost that much since the use of mechanical mixers began requiring their replacement after seven or eight years. Before the mixers were installed, their life was more than twice as long. Use of mixers is believed to help increase the corrosion rate. This type mixing is less time consuming than the old oil circulation turnover method, thus allowing an increased oil batch throughput. Agitation, being more violent than the oil circulation method, removes more of the protective corrosion scale build-up by its washing action from the upper half of the shell, thereby allowing more rapid corrosion. Relatively rapid corrosion also occurs along the center one-third of the steel columns supporting the tank roof. This corrosion is considered to be a result of descaling by a rather violent washing action of the liquid during mixing operations.

Figure 6 is a close-up photograph taken after four and one-half years[3] of service of an uncoated top six-foot ring from a 140-foot diameter by 40-foot height cone-roof light oil blending tank recently dismantled. This tank was equipped with mechanical mixers. Original thickness of

Figure 4—Catalyst-cured epoxy-modified phenolic coating on interior of demineralized water storage tank after more than 2 years of service.

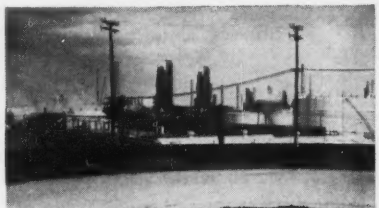

Figure 5—Interior of an uncoated tank for storage of treated water.

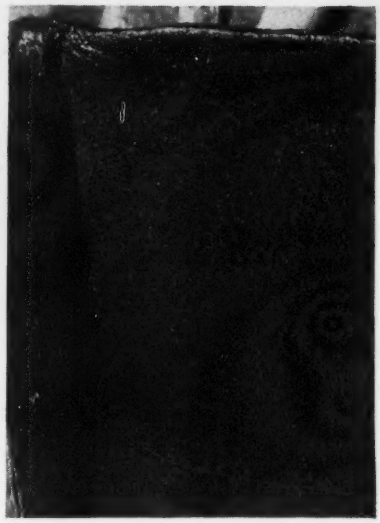

Figure 6—Close-up view of uncoated top ring and angle of a light oil blending tank after 4½ years of service.

the ring was 0.250 inches. Micrometer measurement at the deepest pits in the top foot showed 0.212-inch metal thickness; below this level, 0.165 inch. Metal thickness at pits in the second six-foot ring down from the top also measured 0.165 inch, a loss of 0.085 from its original 0.250 inch, equalling 0.019 inch or 19 mils per year. This corrosion rate indicates that these top rings would have to be replaced after seven to eight years of service.

Interior roof support columns suffered deep pitting and perforations and in one case reached a seriously weakened condition nearing collapse after 17 years of service, the last 12 of which were after installation of mechanical mixers.

When the fact was realized that this interior corrosion problem could affect 30 or 40 large tanks, experimentation was begun to effect a remedy. In early 1954, the tank shell's upper half and entire length of the roof support columns of a new 120-foot diameter by 48-foot height tank were standblasted to a white metal finish. After weld splatter was removed, all weld seams and sharp metal points were ground to rounded surfaces before two coats of a catalyzed epoxy-modified phenolic coating were cross-spray applied to a 10 to 12-mil film thickness.

Figure 7 shows a view of this coating application. The two abutted channels forming the roof support columns had been spot welded, leaving a slight void. These voids along the channel abutments were filled between coats by caulking gun application with a bead of catalyzed epoxy-modified phenolic caulking compound. A concrete bottom was poured over the steel bottom after coating work was completed. Slight streaking that appears in the photograph is a result of moisture from concrete curing condensing on the coating surface and running down through accumulated dust from sandblasting the columns, which was done after completion of the shell coating work. Four other large light oil blending tanks have been coated similar to the first. Although for a period of four years none of these tanks could be taken out of service for coating inspection, other service results of the coating in contact with light oils justified more applications.

The first tank coated was inspected in May, 1958, after continuous light oil blending service since March 1954. Approximately 90 percent of the coating was intact and unaffected. There were only widely scattered small corrosion spots on the coated portion of the shell, where galvanic cell corrosion apparently had occurred at pin-holes in the coating. Larger coating breaks were found along some of the columns, mostly along the edges of the channels and at horizontal welds. There was practically no corrosion creepage underneath the coating at the corrosion spots which comprised only 8 to 10 percent of the coated area. Pit depth at the small corrosion spots on the shell's coated portion varied from 1/16 inches to 3/32 inches. Caulking compound that had been applied to fill the channel abutment voids of the roof support columns was unaffected. Heavy corrosion scale and a high metal loss were found on the upper two-thirds of the uncoated bottom half of the shell.

This coating was considered very good and economically worthwhile protection. Repair of the coating by spot sandblasting and spot coating and sandblasting and coating the remainder of the tank shell with the same material were considered to offer an economic advantage. Future applications will be checked more closely for pin-holes.

Cooling Tower Fan Assemblies

Corrosion of metal portions of the air draft fan assemblies at water cooling towers has been a serious problem. These are exposed to a constant high humidity fog atmosphere from chemically treated water circulating through the tower. Although the metal parts are coated by the manufacturer, coatings are usually too thin for long-term protection. In some cases, new fan assemblies required additional coating before installation because rust along edges had developed in transit. Apparently, baked phenolic thin film coatings had been applied after cleaning the surface by wire brushing and sandpapering. The coating build-up was from one or two mils to about six mils in one coat by application of catalyzed epoxy-modified phenolic coatings.

Figure 8 shows recent photographs of a refinery coated, induced draft fan assembly at one of the Port Arthur refinery cooling towers. Although originally applied by the manufacturer, the coating failed and corrosion occurred after about three years. During a shutdown, the assembly was sandblasted; perforations and pits in the aluminum alloy fan blades were filled with catalyzed epoxy-modified phenolic caulking compound. The framework, hubs and blades were sprayed with two coats of catalyzed epoxy-modified phenolic paint coating to a 6 to 8-mil film thickness. As shown in Figure 8, this coating still gives excellent protection after three years.

Guard Against Product Contamination

This modified epoxy coating has been used successfully as a lining for tank cars in transformer oil transportation service. This highly refined oil must meet a rigid dielectric strength test; no contamination can be tolerated. This coating, spray applied in one coat to a film thickness of 3 to 4 mils to two car interiors after rotary wire brush surface preparation, is currently in good condition after more than three and one-half years' service. Coating of these car interiors greatly facilitate cleaning prior to filling and results in a saving of cleaning labor. The coating also has been used successfully as a lining for interiors of small corrosion inhibitor charge pots at process units to prevent contamination of the inhibitor.

Glaze Coating for Unglazed Tile

Hollow tile interior walls were used in several new buildings at the Port Arthur refinery. These included a bath house, testing laboratory, package warehouse and instrument shop building. In such areas as bath house shower rooms, glazed tile was installed, but in less severe exposure areas such as the bath house locker rooms, laboratory sample retaining rooms, instrument shop work rooms, etc., unglazed tile was installed as an economy measure.

Since the unglazed surfaces are subject to absorption of atmospheric and other

contact contaminants or soil agents, these surfaces are difficult to clean and/or keep clean for a good appearance. Experimentation indicated that one of the catalyst-cured epoxy-modified phenolic clear coatings, applied first to a small area of unglazed tile as a mist spray coat and followed shortly by a heavier, cross-spray coat to a 3 to 4-mil film thickness, would give a glaze surface impervious to absorption, and thereby prevent unremovable soiling.

All unglazed surfaces were coated with this clear coating. After more than three years, the coating is in excellent condition. Cost of the unglazed tile plus that of the coating application is about three-fourths the cost of glazed tile. In the coating operation, the mortar also is given glaze protection.

Concrete Deck Waterproofing

At the Port Arthur refinery in 1955, leakage from rainfall was entering the control room ceiling of one process unit, apparently from slight cracks in the concrete decking surface above, which served as an exchanger platform. Several unsuccessful attempts to stop this leakage included wedge excavation along the cracks, coating these with bituminous materials and refilling with cement grout. A large deck section was cleaned with a chemical solution, neutralized, primed with a thinned penetrating coat of clear epoxy-modified phenolic and finish coated with two 4-mil coats of pigmented catalyzed epoxy-modified phenolic. Dust-free, fine-grained silica sand was sprinkled into these finish coats during the cross-spray application to make the surface skid-proof; this deck is a working area during unit turnaround and a walkway area during normal operations. During turnarounds, this floor surface is subject to oil spillage and abrasion during exchanger cleaning, thereby making oil-soluble and soft coatings inadvisable.

After more than three years since coating application, no leakage into the control room has occurred.

High-Film-Build Alkyds

In 1953, one manufacturer claimed that a new formulation alkyd type white tank paint could be cross-spray applied by multiple pass in one coat to film thicknesses several times that of the conventional alkyd type without resulting in runs, sags or skin-dry wrinkling. With most of the conventional whites, film thicknesses of about 1½ to 2 mils per coat were accepted standards. Because these thicknesses usually did not give good hiding, two finish coats were normally specified.

As a result of extensive literature studies and accumulated data on light oil vapor losses, large gasoline tanks at the Port Arthur refinery were painted white. Two small and three large tanks were finish coated, after priming, with two coats of the conventional alkyd type white finish. These were spray applied at a spreading rate of approximately 250 sq ft per gallon per coat, considered the lowest numerical rate at which these could be applied without resulting in runs or skin-dry wrinkling. This gave finish coat film thicknesses of 3 to 4 mils and resulted in good hiding.

Early in 1954, the first one-coat, multi-pass spray application of high-film-build white was made at an average spreading rate of 140 sq ft per gallon. This resulted in good hiding without any runs or sags. Average film thickness of this coat was 4 mils with the thickness ranging between 2 and 5 mils caused by the effect that variable wind and other conditions had on the amount deposited.

Since this initial application, 17 similar one-coat finish applications of high-film-build alkyd paints have been made after conventional surface preparation and priming, to tanks ranging in size from 120-foot diameter by 40-foot height to 160-foot diameter by 48-foot height. However, special efforts were made to obtain a 150 to 160 sq ft per gallon spreading rate to produce a dried white film of 3.5 to 4.0 mils. Experience proved that a film thickness of more than 4 mils resulted in top film mud cracking after about one year's weathering.

Experimental multi-pass cross-spray one-coat applications of so-called conventional alkyd finishes primarily recommended by the manufacturers for two-coat applications were tried at spreading rates of 150 to 160 sq ft per gallon. These gave satisfactory hiding and films of 3.0 to 3.5-mil thickness without runs or skin-dry wrinkling when applied during atmospheric temperatures above about 80 F. When applied at lower atmospheric temperatures, low spreading rate, high film thickness and satisfactory hiding were difficult to obtain in one coat without runs because of slower solvent evaporation. Of 10 one-coat finish applications of conventional whites made on larger size tanks, only two were applied at atmospheric temperatures that made film application difficult.

Recently, multi-pass spray application of a high-film-build alkyd red lead primer was made to the sandblasted surfaces of one small and several large new steel cone-roofed tanks at the Port Arthur refinery. On the small tank, one coat of primer was applied at an average spreading rate of 128 sq ft per gallon, resulting in a 6 to 8-mil dry film without running. The large tanks received two primer coats; high-film-build alkyd red lead was applied there at a 200 to 250 sq ft per gallon spreading rate to obtain a 2 to 3-mil dry film without runs.

Summary and Conclusions

Developments in coating formulations and application techniques during the past six or seven years permit field application of thick films of synthetic coatings. Equipment out-of-service time can be reduced because necessary film thickness is obtained in fewer coats. This is possible because of hot-spray application, catalyst-curing coating formulations and sprayable high-film-build alkyd paints.

Field test applications of these materials show that satisfactory protection can be obtained for oil refinery equipment surfaces such as the following: metal surfaces at waste water separators, cooling tower

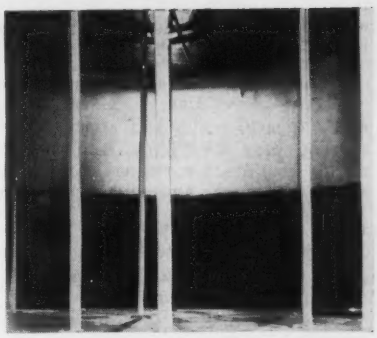

Figure 7—Interior of light oil blending tank. Top half of the interior and the columns have been coated with a catalyst-cured epoxy-modified phenolic coating.

Figure 8—Cooling tower fan assembly with catalyst-cured epoxy-modified phenolic coating. Assembly has been in service approximately 3 years.

piping, interiors of exchanger shells and channels, tanks interiors and exteriors, cooling tower fan assemblies and masonry walls.

For best results, good surface preparation is of primary importance. As with the conventional vinyls and baking type phenolics, a good sandblast anchor pattern of about 2-mil depth blasted to white metal and a dry dust-free surface are required to obtain good results from hot spray vinyls and catalyst-curing coatings. For interior applications, use of vacuum equipment for removal of sandblasting sand and dust particles is advisable. Success or failure of the coating generally depends on attention or inattention to certain techniques of application. Therefore, close inspection should be maintained during surface preparation and coating application to see that the coating manufacturer's recommendations are followed. The coating should be cured adequately before process service is begun.

A special technique used during coating application to tanks interior has been to sandblast and apply all coats to the upper portion; then, the bottom is sandblasted and the first coat brushed on. This eliminates fall-out of dry overspray onto the bottom, minimizes imbedding of sand and dust particles into the coating insures better adhesion and durability.

Hot spray vinyls apparently eliminate one of the disadvantages encountered in refinery usage of conventional vinyls: requirement of multiple coats to provide adequate pinhole-free film thickness. Although the hot spray type still has the limitation of being subject to blistering at temperatures above about 150 F, long-term flexibility of the vinyl type indi-

cates that it should protect alternating wet and dry surfaces such as the exterior of piping adjacent to cooling towers and those at waste water separators at temperatures below 150 F. Resistance of vinyls to acid and alkali attack makes the hot spray thick films useful as protective coatings for refinery equipment subjected to acid and alkali exposures.

At the Philadelphia refinery thick-film applications of catalyst-cured phenolic coatings have protected exchanger channels and heads. Catalyst-cured epoxy caulking compound prevents "weepage" at riveted tank seams in certain chemical solution services.

At the Port Arthur refinery, thick-film applications of catalyst-cured epoxy-modified phenolic coatings have protected surfaces exposed to low concentrations of sulfuric acid, to tank interiors in propane and other light hydrocarbon service, to vessel interiors in water service including exchanger shells, channels and heads and steam condensate tanks with condensate temperatures about 180 F. These coatings are resistant to high concentrations of caustic soda solutions and solutions of oils and caustic soda at temperatures to 200 F and probably higher. This type of thick-film coating has given satisfactory service on equipment in warm, high humidity atmosphere such as that of fan assemblies at refinery water cooling towers. It has been used successfully as a coating for tank car interiors as a guard against contamination of highly refined oils and as a protective glaze coating for unglazed tile. It has also been used successfully as a pore sealer and waterproofing coating.

Multi-pass cross-spray applications of high-film-built alkyd white finishes, in one coat, to properly primed oil storage tanks at the Port Arthur refinery have shown that, by meticulous application, dry films to 4-mil thickness are as satisfactory as conventional alkyd whites applied in two complete coats. However, film thickness higher than 4 mils results in "mud cracking" after about a year of exposure. Application of two complete coats, each of about 2-mil dry film thickness, requires less experienced technique and is more fool-proof. Experimental spray applications of high-film-build alkyd red lead primers to sandblasted steel tanks indicate that satisfactory one-coat applications of these may be made to vertical surfaces at dry film thicknesses as high as 6 to 8 mils.

References

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Any discussion of this article not published above will appear in the December, 1959 issue

TECHNICAL PAPERS ON CORROSION WELCOMED

Authors of technical papers on corrosion are invited to submit them for review without invitation to the Editor of Corrosion. Write for "Guide for the Preparation and Presentation of Papers" sent free on request to prospective authors.

Corrosion Rate Measurement by Hydrogen Effusion In Dynamic Aqueous Systems At Elevated Temperature and Pressure*

By M. KRULFELD, M. C. BLOOM and R. E. SEEBOLD

Abstract

A method of applying the hydrogen effusion method to the measurement of corrosion rates in dynamic aqueous systems at elevated temperature and pressure is described. Data obtained in low carbon steel systems are presented, including (1) reproducibility obtained in measured hydrogen effusion rates at a flow velocity of 1 foot per second at a temperature of 600 F and 2000 psi, and (2) a quantitative comparison between the hydrogen effusion rates in static and in low flow velocity dynamic systems at this temperature and pressure.

Some observations are included on corrosion rate measurements in a high flow velocity (30 feet per second) loop by the hydrogen effusion method. Implications of these measurements with regard to the comparison between high flow velocity corrosion and low flow velocity corrosion are mentioned and some data indicating high local sensitivity of the hydrogen effusion method are noted. Some possible difficulties involved in the method are pointed out. 2.3.4

Introduction

A HYDROGEN effusion method for corrosion rate measurements in aqueous systems at elevated temperature and pressure has been described previously[1] and data obtained by its application to the corrosion of mild steel in static systems have been published.[2] The method is based on the fact that in a sealed metal container, at elevated temperature, hydrogen evolved as a result of the corrosion reaction

$$\text{Metal} + H_2O \rightarrow \text{Metal Oxide} + 2H \quad (1)$$

will permeate the walls of the container at a comparatively rapid rate. It is possible therefore to calculate corrosion rates from measurement of the rate of hydrogen effusion through the walls of sealed vessels which are being corroded by the water or the aqueous solutions which they contain.

For the purpose of these measurements, the sealed vessels of the metals under test are surrounded by an evacuated system of known volume. The rate of hydrogen effusion is calculated from measurements of the rate of pressure rise in the evacuated system. It is known that in dynamic systems the fluid flow velocity within piping and around the corroding metal components of such systems can have a considerable effect on the rate of corrosion.[3,4] For this reason it was thought desirable to test the applicability of the hydrogen effusion method to corrosion measurement at elevated temperature and pressure in dynamic systems. This paper describes results obtained in this connection. No attempt was made to surround the entire dynamic system with an evacuated space as in the case of the sealed containers of the static system above mentioned. Instead, local sections of the dynamic system were surrounded by evacuated spaces and the rate of hydrogen build up in these spaces was measured.

Experimental Apparatus and Procedures

Each dynamic system consists of sections in which vacuum-tight cans have been welded around the steel piping through which the high temperature aqueous solutions are circulated. Hydrogen resulting from the corrosion reaction within these pipe sections effuses through the hot pipe wall into the evacuated can surrounding the pipe. The rate of hydrogen pressure rise within the can is recorded by a Pirani gauge circuit, and from these data, combined with the volume and temperature of the gas collecting system, the rate of hydrogen effusion is calculated. This, combined with the surface area of the pipe enclosed by the can, and the stoichiometry of the reaction

$$3\,Fe + 4H_2O \rightarrow Fe_3O_4 + 4H_2 \quad (2)$$

permits calculation of the corrosion rate of the pipe wall corresponding to this hydrogen effusion rate.

MYER KRULFELD is on the consultant's staff on corrosion of the Metallurgy Division, Naval Research Laboratory, Washington, D. C. He received a BS in chemistry from Worcester Polytechnic Institute (1946) and a PhD in physical chemistry from the University of Illinois (1951). He has been with the Metallurgy Division of NRL since 1951.

M. C. BLOOM is consultant in charge of corrosion research in the Metallurgy Division of the Naval Research Laboratory, Washington, D. C. He received a BS in chemical engineering from MIT in 1922. Six years in the paper and petroleum industries were followed by a return to MIT where he obtained a PhD in physical chemistry and worked as a research associate in crystal chemistry and electrochemistry. In 1949 he joined the Naval Research Laboratory as a consultant after several years of private consulting.

ROBERT E. SEEBOLD is in the Optics Division at the U. S. Naval Research Laboratory, Washington, D. C. After joining NRL's Metallurgy Division in 1954 he worked on corrosion problems in the fields of liquid metals, molten salts and water corrosion as related to nuclear reactor applications. Prior to this he participated in the sodium evaluation program conducted by the Chemistry Division at NRL where he co-authored several papers on the physical-chemical properties of liquid metals. He received a BS in chemistry from The George Washington University and is an active member of ACS and NACE.

Thermal Convection Loops

The dynamic systems used in this work consisted chiefly of thermal convection loops of SAE-1010 ½-inch schedule-80 pipe, a schematic diagram of which is given in Figure 1. Each thermal loop was 31 inches wide and 53 inches high and consisted of flanged sections as shown. As indicated, three of the flanged sections of pipe had vacuum-tight cans welded around them for the collection of effused hydrogen gas from the corroding inside surface of the pipe at temperature.

Each can was connected by means of one vacuum valve to a common vacuum manifold and pumping system, and by means of another vacuum valve to a Pirani bulb and gauge circuit which was a modification of that of Schwartz and Lavender.[5] The Pirani circuit output was recorded. The heater indicated at the bottom of the loop was an external resistance heater controlled by a Brown Pyrovane controller to give the desired temperature to the water circulating in the loop. In the cooler, copper tubing through which cooling water was passed was wrapped around the outside of the pipe.

The surge tank was a cylinder fabricated from 3-inch schedule-80 low-carbon-steel pipe, 18 inches long, connected to the loop by ¼ inch OD, ⅛ inch ID low-carbon-steel tubing. Another external resistance heater, Pyrovane controlled, was used to fix the temperature of the surge tank. In use, the loop system was filled with water and the surge tank partially filled. This permitted the surge tank to take up any hydrostatic expansion due to temperature variations in the loop. By maintaining the temperature of the surge tank above the temperature

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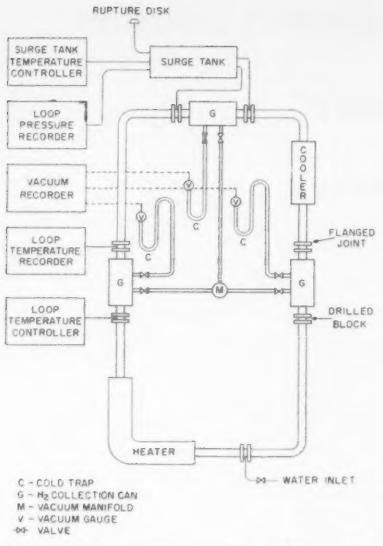

Figure 1—Schematic diagram of thermal convection loop.

of the loop it was also possible to maintain the pressure of the system at a level (2000 psi) which prevented boiling in the loop (at 316 C). The system pressure was recorded and a control was provided to cut off the heaters should the temperature control fail and the pressure become excessive. A rupture disk also was provided as a safety device should the other controls fail.

Preparation and Assembly of Loop Parts

The thermal convection loops were fabricated from ½-inch SAE-1010 schedule-80 steel pipe. Prior to assembly, all inside surfaces of the piping were cleaned by treatment with 2.3 percent H_2SO_4 at 85 C for approximately 10 minutes, followed by prolonged rinsing with distilled water to remove all traces of acid. The canned sections of pipe required special treatment due to the need for special cleanliness within the evacuated can section. This was done in order to avoid excessive outgassing from the system during hydrogen gas collection.

Figure 2 is a photograph of a canned length of pipe which has been cut in half to show details of construction. After the can was welded to the pipe, vacuum valves were in turn welded to the two side connections on the can. The can and enclosed pipe were then placed in a special furnace which allowed the side connections from the can to project outside the furnace. Hydrogen gas, deoxidized and dried by passage through a DeOxo Hydrogen Purifier (Baker and Company, Newark, N. J.) and a liquid nitrogen cold trap, was passed for ½ hour through the gas collection chamber. The chamber was maintained at a temperature of 675 C, in order to reduce most of the oxides and other reducible impurities present to a minimal value. The hot can was then evacuated until the pressure became less than 20 microns (approximately 15 minutes) after which the can was allowed to cool in the furnace under vacuum. The inside surface

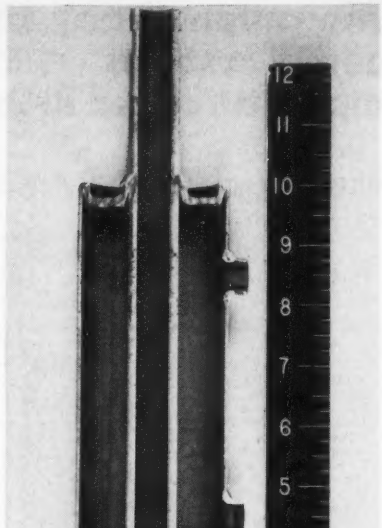

Figure 2—Section through pipe with attached can for effused hydrogen collection.

of the pipe was then cleaned with hot 2.3 percent H_2SO_4 in the same manner as all other loop sections.

Loop assembly was by means of flanges drawn up against SAE-1010-steel blocks and sealed with stainless steel Flexitallic gaskets (Flexitallic Gasket Company, Camden, N. J.). The blocks were drilled out to a diameter equalling the inside pipe diameter and were used for connections to the interior of the loop by drilling holes into the sides of the block and welding tube or pipe connections into the hole. Connections to the surge tank or to pressure valves for sampling or admission of fluid were made in this manner and wells for the insertion of thermocouple wires were also welded into such holes in the block. After assembly the tightness of each loop was checked by pressurizing with an inert gas. When tight, the gas normally was removed from the loop by evacuating the system. After this the aqueous fluid to be used for the run was admitted and the pressure valves used for evacuating and filling the loop were closed, the heaters for loop and surge tank were turned on, and water flow was started through the cooling coil. Hydrogen effusion became appreciable at a threshold temperature of about 148 C.

Measurement of Corrosion

Measurement of corrosion rates by hydrogen effusion depends upon accurate measurement of the amount of hydrogen effused over a given time period. This in turn involves knowledge of the pressure, volume, and average temperature of the gaseous hydrogen collected.

As can be seen from Figure 1, the volume of each gas collecting system included, in addition to the can volume the volume of the connection from the can to the vacuum valves and from one vacuum valve to the vacuum gauge. This latter connection was 5/16-inch-OD-copper tubing attached to the valve extension and to the Pirani bulb by flanges and neoprene o-rings to ensure tightness. The U-bend shown in the tubing was used as a cold trap. The volume of each system was determined from its known geometry and was checked by expansion of a known volume of gas into the system at room temperature. The volume was found to be 550 ± 5 cm^3.

The pressure within the system was obtained from the Pirani circuit record. The Pirani gauge was calibrated with dry hydrogen gas and a McLeod gauge prior to each run to ensure accuracy. Chilling of the cold trap with liquid nitrogen during measurement ensured that no pressure due to the outgassing of any condensable gases from the system would be measured.

Determination of an average temperature for the hydrogen gas collected was somewhat difficult, due to the varied temperatures of different parts of the system. However, by expansion of a known amount of dry hydrogen gas into the evacuated collecting system, and applying the gas laws, it was possible to calculate that the average temperature of the hydrogen gas in the collecting system was approximately 105 C. During the expansion process the loop was maintained at 316 C and the cold trap was chilled to simulate the exact conditions encountered during an actual measurement of hydrogen effusion due to corrosion. The hydrogen effusion rate obtained from the above data was combined with the stoichiometry of the reaction $3Fe + 4H_2O \rightarrow Fe_3O_4 + 4H_2$ to calculate the equivalent weight of steel corroded. Knowing the surface area of the corroding pipe within the gas collecting can (a 6.5-inch length of ½-inch-ID pipe) the corrosion rate per unit area could be obtained. In this way it was possible to calculate that a pressure rise within the gas collecting system of 1 micron of hydrogen per minute was equivalent to a weight-loss corrosion rate of 56 milligrams per square decimeter per month (mg/dm^2/mo) within the pipe.

Flow Velocity

An indication of the rate of flow within the thermal convection loops was obtained by injecting 5 to 10 cm^3 of cold water into a loop by means of a Sprague high pressure pump and following the flow of the cold water through the loop by means of thermocouples in the stream. Due to diffusion of the cold water into the hot, and the rapid warm-up of the small volume of colder water the precision obtained was not high. However, a series of experiments in a loop with a hot-leg temperature of approximately 316 C and a cold-leg tem-

perature of approximately 305 C, at a pressure of 2000 psi, indicated that the flow velocity under these conditions was of the order of 1 foot per second.

Experimental Results

Initial results in the thermal loops were erratic and reproducibility between the corrosion rates measured by three hydrogen effusion systems on the same loop run at 316 C tended to be poor. Most of the difficulties proved to be a result of small leaks in the vacuum cans and associated apparatus used to measure the amount of hydrogen effusing from the loop piping. When the leaks were eliminated fair reproducibility in the corrosion rates measured at the three separate canned sections on the same loop were obtained. The worst reproducibility obtained in tested vacuum-tight systems is shown in a run in Figure 3. The arithmetic mean of the values obtained in the three measuring systems is shown in the graph, with the individual measured values indicated. The maximum deviation from any of the mean values in this run was ± 26 percent. No greater deviation from the mean value was found in any run using systems leak-tested at temperature (316 C).

The use of hydrogen effusion measurements to calculate corrosion rates is based on the assumption that the gas whose pressure is measured is pure hydrogen. In order to check this, the non-condensable gas from two measuring systems was transferred to a gas analysis apparatus, where hydrogen was determined by reaction with cupric oxide at 325 C to form water. Of 270 microns of gas pressure 260 microns were shown to be due to hydrogen, indicating a hydrogen content of 96.3 percent in the gas. Since some contamination of the gas undoubtedly occurred during transfer and handling in the analytical process, the actual percentage of hydrogen in the cold-trapped gas in a measuring system as used is indicated to be close to 100 percent.

Two experiments were performed to check whether the rate of hydrogen effusion into a welded vacuum can was controlled primarily by the corrosion and effusion processes taking place at the pipe wall to which the can is welded, or to other factors such as hydrogen concentration in the circulating loop water. A loop was assembled in which two of the canned pipe sections had been pretreated for 27 days in a loop operated at 316 C with distilled, deionized, degassed water, thus reaching an indicated corrosion rate of the order of 30 mg/dm²/mo. The interior of the third canned pipe section had been freshly cleaned prior to loop assembly. When the run was made in this loop at 316 C using a fresh charge of distilled, deionized and degassed water, the corrosion rates indicated by the pretreated pipe sections continued low (of the order of 21 mg/dm²/mo after 1 day and 9 mg/dm²/mo after 27 days). The corrosion rate indicated in the freshly cleaned pipe section was initially much higher (410 mg/dm²/mo) and followed the typically decreasing rate curve observed in a new loop prepared with clean pipe sections (see Figures 3 and 4), reaching a rate of 29 mg/dm²/mo in 27 days. The indication was that hydrogen effusion rates depend primarily on the local pipe wall corrosion process.

In the other experiment, 50 cm³ of hydrogen gas at normal temperature and pressure were injected into a loop during a run by means of a bomb equipped with high pressure valves. Information regarding the hydrogen gas concentration normally present in loops as a result of corrosion under the conditions of these experiments is not available, but evidence of such concentrations under related conditions[6] indicates a probable concentration of the order of 10 cm³ hydrogen per liter. The 50 cm³ injected into the 2900 cm³ of the loop plus surge tank volume provided an additional hydrogen gas concentration of 17 cm³ per liter. No change in the indicated corrosion rate was observed as a result of the hydrogen addition to the loop water, again indicating dependence of the hydrogen effusion rate upon local pipe wall corrosion, rather than on hydrogen concentration in the circulating high pressure water.

The corrosion rates calculated from a series of five runs using pure water at 316 C are shown in Figure 4. Three of the runs were made using distilled water treated with deionizer resin (Amberlite MB-1) to remove any remaining ionized impurities. The distilled water was boiled in a stainless steel reflux apparatus to eliminate any air in solution, and stored under helium gas in a polyethylene container during transfer to the loop.

Two runs were made with distilled air-saturated water stored in polyethylene containers prior to use in the loop. The results do not show a clear correlation between the two different types of water used. The corrosion rates measured (arithmetic mean of three measuring systems for each run) indicate a wide variation from run to run in the first few days. The rates from all runs approached each other after two weeks and almost coincided for three of the five runs at that time. As the runs continued the rates became more and more similar, and extrapolation to 35 days of all of the runs would appear to give essentially similar corrosion rates for all five of the loops at that time.

The lack of reproducibility of rates measured in the first few days may be due either to: (1) Variation in the actual corrosion rates (which might initially be strongly affected by slight differences in the pipe surfaces, a factor which would become less important as corrosion product films covered the initial surface) or, (2) Lower reliability in the hydrogen effusion method in the early portions of a loop run. It is clear, however, that agreement between the results obtained from run to run improves as the corrosion time is extended and is reasonably good for this type of data after a month.

Run 21E, one of the five included above, was made for special comparison with runs simultaneously made in static capsule systems[1] using identical water and low carbon steel of similar composition. The water used in capsules and in the loop was taken from the same polyethylene container into which it had been distilled. The water was stored for seven days in the container prior to use both in the loop and in the capsule runs. An additional amount of water from the same still batch was distilled into a platinum container and stored there prior to use in another series of capsule runs.

The results of loop run 21E and of the two series of capsule runs are shown in Figure 5. In one capsule run, water identical to that in the loop run was used; in the other the water used was identical except for storage in platinum instead of in polyethylene. Experience with other runs in capsules indicates that the slightly lower corrosion rates shown by one of the above sets of capsules at 80 days may be anomalous, and that rates more closely approaching those shown by the loop run and by the other set of capsules run at this time may normally be expected.

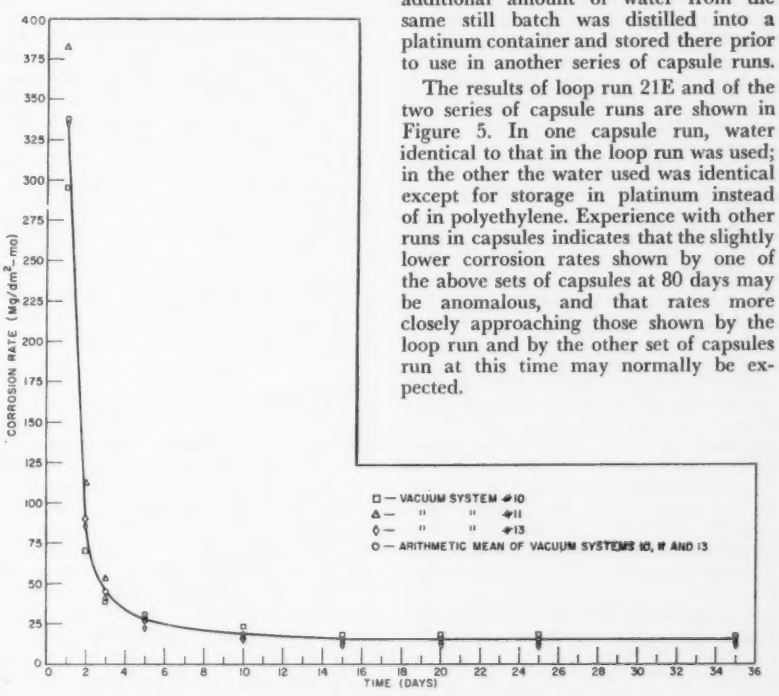

Figure 3—Reproducibility of corrosion rate measurements by different vacuum systems on a thermal convection loop.

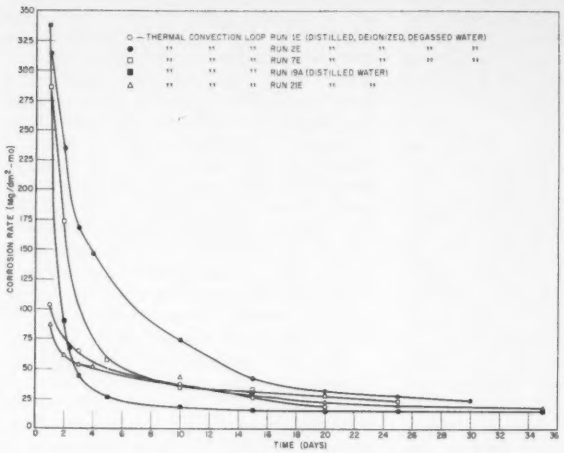
○ — THERMAL CONVECTION LOOP RUN 1E (DISTILLED, DEIONIZED, DEGASSED WATER)
● " " " RUN 2E " " " "
□ " " " RUN 7E " " " "
■ " " " RUN 19A (DISTILLED WATER)
△ " " " RUN 21E " "
CORROSION RATE (mg/dm² - mo)
TIME (DAYS)

Figure 4—Comparison of corrosion rates measured in different thermal convection loop runs.

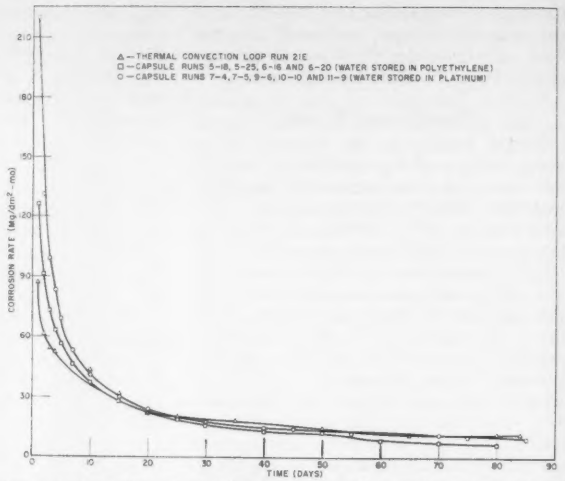
△ — THERMAL CONVECTION LOOP RUN 21E
□ — CAPSULE RUNS 5-18, 5-25, 6-16 AND 6-20 (WATER STORED IN POLYETHYLENE)
○ — CAPSULE RUNS 7-4, 7-5, 9-6, 10-10 AND 11-9 (WATER STORED IN PLATINUM)
CORROSION RATE (mg/dm² - mo)
TIME (DAYS)

Figure 5—Comparison of corrosion rates measured in static and in dynamic systems by hydrogen effusion.

Some Observations on a High Flow Velocity Loop

In addition to the work in thermal loops reported above, a few runs have also been made in a larger loop with a liquid flow velocity of 30 feet per second. The loop used was similar in construction to the thermal loops, except that flow was effected by means of a stainless steel pump with a canned motor (Westinghouse 150C pump) instead of by thermal convection. One and one-half inch schedule-80 SAE-1010-steel pipe was used throughout, the height of the loop being 40 inches and the length 72 inches. The volume of the vacuum measuring systems and the surface area of the canned pipe were also different from those in thermal loops. For this reason a different factor was used for calculating corrosion rates from rate of hydrogen pressure rise (1 micron per minute of pressure rise = 25 mg/dm²/mo of weight loss for the pumped loop, compared with 56 mg/dm²/mo for the thermal loops). Provision was made in this loop for the insertion of corroding specimens into one of the canned pipe sections.

The runs made in this apparatus were too few and the results not sufficiently reproducible to warrant reliable quantitative conclusions regarding corrosion rates at the flow velocity of 30 feet per second. Some of the results obtained, however, appear of interest.

All the corrosion rates measured, compared to corrosion rates in thermal loops after a similar time interval from the beginning of a run, were much higher in the high velocity loop. After 14 days, for example, the corrosion rates observed in the pumped loop, for different runs, were 2.5 to 9 times the average of those found in thermal loops. This indicated that the carbon steel corrosion rate at a flow velocity of 30 feet per second in 316 C water at 2000 psi is considerably higher than the corrosion rate in similar water at a flow velocity of 1 foot per second.

In one run, a sample holder with corrosion specimens (longitudinal segments of the same piping from which the loop was made, 4½ inches long, ¾-inch wide, and 3/16-inch thick) was placed in one of the three canned sections while the other two sections were without specimens. The total surface area of corroding metal in the pipe section containing specimens was 2.3 times the corroding metal area in the empty pipe sections. The hydrogen effusion rate from the pipe section containing the specimens was found to be two to three times that from the empty pipe sections. The ratio was followed with fair consistency over the entire course of the run, over widely varying corrosion rates as the run proceeded (95 to 305 mg/dm²/mo for sections without specimens, 280 to 690 mg/dm²/mo for the section with specimens).

The higher effusion rate from the section containing specimens undoubtedly was due to corrosion of the specimens and holder within the section, added to the normal corrosion occurring at the pipe wall. These results indicate the high sensitivity to local corrosion of the hydrogen effusion method. The method appears to be affected by corrosion occurring near the pipe wall through which the measurement was being made, as well as by corrosion of the pipe wall itself.

Some optical observations on corrosion in the high flow velocity loop are being presented in a separate article.[7]

Discussion

The close agreement between the results obtained in the static test and those obtained in the dynamic test at about 1 foot per second and the pronounced sensitivity to local changes in conditions at the pipe wall encourage the belief that useful data may be obtained by this application of hydrogen effusion measurements to dynamic systems. It must be pointed out, however, that, in contrast with the apparatus used in the static case, the apparatus used in the dynamic case may introduce hydrogen into the corroding area. Such hydrogen may be introduced by transfer through and with the moving fluid from sources other than the local corrosion which it is desired to measure. Likewise the apparatus used in the dynamic case may remove from the corroding area some of the hydrogen therein generated by the corrosion reaction. Whether this is a compensating effect and thus does not influence the reliability of the measurement has not been adequately explored.

It has been shown that corroding specimens inside the high velocity loop increased the rate of hydrogen effusion from the pipe wall in the immediate vicinity of the specimens. This then raises the question as to whether this hydrogen effused directly into the pipe wall through the metallic contact between the specimen holder and the pipe wall or whether it was generated originally as molecular hydrogen which so increased the local concentration of molecular hydrogen as to cause the observed increase in effusion. Further data, perhaps using corroding samples insulated from the pipe wall, and checking sample corrosion rates by weight loss for correlation with pipe wall corrosion rates by hydrogen effusion are required for proper establishment of the reliability of the method for quantitative purposes.

Acknowledgments

Particular thanks are due to F. A. Brandt who assembled much of the apparatus employed in the early phases of this study and to W. S. Pellini who suggested the method employed in measuring flow velocity in the thermal loops.

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Visual Observations on the Water Stream In An Elevated Temperature High Velocity Loop*

By R. E. SEEBOLD*

Abstract

The role of oxygen in the corrosion associated with high temperature water systems utilizing low carbon steel containment has been under study for many years. This report describes the "in situ" visual effects of oxygen and oxide coatings on the corrosion occurring in a high velocity water loop system at temperatures up to 600 F. The visual effects are correlated with previously reported corrosion data measured by physical methods. It is concluded that oxygen inhibits the corrosion process. 3.4.6

Introduction

IN A PREVIOUSLY reported corrosion study[1] it was indicated that:

1. The measurement of corrosion by the method of hydrogen effusion seemed to have a threshold temperature of 150 C.

2. When the loop charge contained oxygen the indicated corrosion at temperatures up to 316 C was extremely low.

In order to further investigate these indications an apparatus was designed for direct visual examination of corrosion and Tyndall effect in a high temperature-high velocity water loop.

Apparatus

The visual observation tube is shown in Figure 1. There are three quartz windows set normal to the water stream as indicated. This positioning allows observation either by back lighting or by 90 degree incident light. The back lighting may be used to measure the relative optical density of the loop stream while the 90 degree incident light is necessary to any study of Tyndall effect in colloidal dispersion. Gasketing of the quartz windows with Flexitallic gaskets allows the entire assembly to operate at the temperature of the loop stream and thus eliminates thermal gradients which could interfere with visual observation.

The loop shown in Figure 2 was composed of 1½-inch-Schedule 80 low carbon steel piping with a total low carbon steel area of 1070-square inch internal surface. Circulation was maintained at a velocity of 30 ft/sec by means of a Model 150-C Westinghouse pump.

Experimental Procedures

The entire low carbon steel section of the loop was cleaned in 2.3 percent sulfuric acid, thoroughly rinsed in distilled water, and dried in reagent grade acetone. The loop was then assembled and the observation section was installed. After evacuation by means of a mechanical vacuum pump to 10 microns the loop was filled with degassed distilled water. A pressure of 2000 psi was applied to the loop charge by means of a heated surge tank connected to the loop through a 1/16-inch ID tubulation. Heating of the loop was accomplished by both the action of the impelling pump and external heaters. Approximately 45 minutes were required to heat the loop stream from 24 C to 316 C. All temperature measurements were made by means of thermocouples contained in wells in the loop stream.

In the first and fourth observation runs the entire loop was cleaned prior to assembly. These two runs differed in that Run No. 1 was made using degassed distilled water while Run No. 4 was made using degassed distilled water to which pure oxygen was added. The gas was added by charging a 10 cc bypass bomb to 2000 psi with pure oxygen at room temperature and then opening the bypass valves to the loop. For Run No. 2 the loop was continued in the oxidized condition resulting from Run No. 1 while for Run No. 3 a portion of the loop amounting to 316 square inches internal surface was cleaned in 2.3 percent acid as previously described and reinstalled in the loop, the remainder of the internal surface remaining in the oxide covered condition.

Results

Table 1 shows the visual observations of changes in loop stream waters during circulation and heating from 24 C to 316 C. Each run covered a total time of

★ Submitted for publication April 11, 1958.
* Optics Division, U. S. Naval Research Laboratory, Washington, D. C.

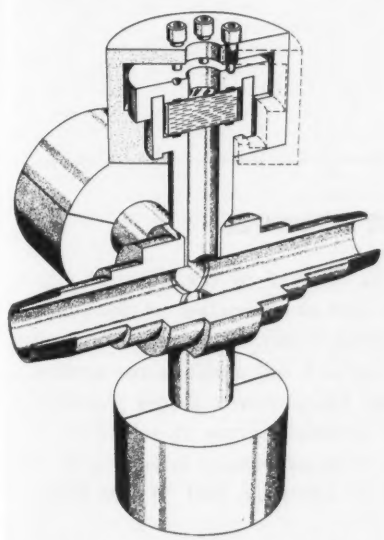

Figure 1—Apparatus for visual observation of corrosion.

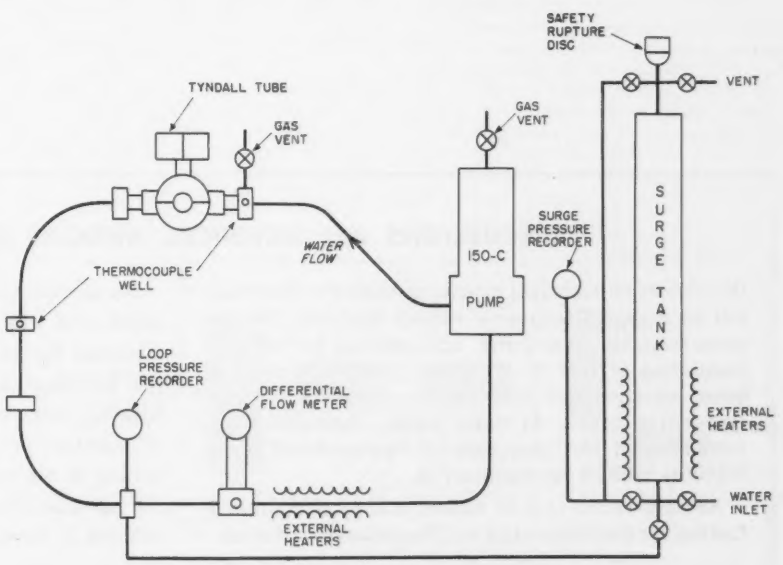

Figure 2—Schematic diagram of high velocity loop.

TABLE 1—Color of Loop Stream Water at Various Temperatures

Temperature, °C	Run No. 1	Run No. 2	Run No. 3	Run No. 4
24–135	Water, clear	Water, clear	Water, clear	Water, clear
+135	Water, yellow	Water, clear	Water very pale yellow	Water, clear
135–163	Water, opaque brown	Water, clear	Water, yellow-brown	Water, clear
205	Sudden clearing	Water, clear	Sudden clearing	Water, clear
316	Water, clear. Solid black particles	Water, clear	Water, clear. Some particulate matter	Water, clear

only one hour of operation. Under no conditions was a Tyndall effect noted.

Conclusions

These experiments show that, visually, the amount of material transported in the stream of a water-charge loop is dependent on the area of unoxidized low carbon steel available to the stream and on the oxyen content of the water charge. The absence of a visible Tyndall effect in all observations would tend to indicate that the material in the loop stream may not be colloidal, although the opacity found in Run No. 1 and the known low solubility of iron compounds in water under the given conditions casts some doubt on such a conclusion.

In the case of Runs No. 1 and No. 2 the stream coloration beginning at 135 C agrees quite well with the previously observed hydrogen effusion threshold[1] This then must be taken as the temperature at which appreciable corrosion begins. The sudden clearing at 205 C can be due only to precipitation of the iron compounds carried in the stream.

In view of previous observations[2] and those in Run No. 4 here, the presence of a proper concentration of oxygen in the water charge would seem to inhibit the corrosion process.

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Any discussion of this article not published above will appear in the December, 1959 issue

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Authors will be sent on request a copy of the NACE Outline for the Preparation and Presentation of Papers.

Discussions will be reviewed by the editor of Corrosion and will be sent to the author of the paper discussed for his replies, if any. Publication will be in the Technical Section with full credit to the authors together with replies. Discussions to papers presented at meetings of the association may be submitted in writing at the time of presentation or later by mail to the editorial offices of Corrosion, 1061 M & M Bldg., Houston 2, Texas.

NATIONAL ASSOCIATION of CORROSION ENGINEERS

Minimum Corrosion For Butane Isomerization Units*

A Contribution to the Work of NACE Group Committee T-8 On Refining Industry Corrosion,[1] By J. F. Mason Jr. And C. M. Schillmoller[2]

NACE TECHNICAL COMMITTEE REPORT

Publication 59—11

Abstract

A review is made of corrosion problems associated with butane isomerization units. In this connection, vapor-phase and liquid-phase butane isomerization are considered separately. Data reported include corrosion rates of nickel and Monel in HCl at 30 C, corrosion of Hastelloy alloys A and B in HCl at room temperature, effect of temperature on corrosion of nickel and Monel in 5 percent HCl, and corrosion of Type 1 Ni-Resist in unaerated HCl at room temperature.

In butane isomerization units it is important that the feed be kept dry and that such impurities as sulfur, pentanes and olefins be reduced to a minimum. Adequate guard chamber capacity is a must. Alloys must be carefully selected for use in locations where corrosion is severe. 8.4.3

Introduction

RELATIVELY LITTLE information has appeared regarding corrosion problems associated with butane isomerization units. This paper will review some of these problems and point out where corrosion has occurred or normally can be expected to occur. Also, remedial steps will be indicated for consideration by those operators who are experiencing severe corrosion in their units.

The increasing demand for aviation gasoline and for motor fuels of progressively higher octane have forced the refiner to make broader use of olefin alkylation with isobutane. The alkylate gasoline has a high neat octane rating, low sensitivity, high lead susceptibility and generally excellent blending properties. The use of alkylate in premium motor gasolines today is considered as an economical means to help meet the high octane demands.

Corresponding amounts of isobutane and olefins (propylene, butylene, amylene and even ethylene) must be supplied. There appears to be no shortage of olefins for the present, although there is a shortage of isobutane. Therefore, there is a decided interest in processes for converting the more plentiful normal butane to isobutane for alkylation. This is part of the trend to further extensive upgrading of the light paraffinic fraction.

Processes

There are essentially two processes that can be applied to the isomerization of butanes to isobutane: the butane vapor-phase process and the butane liquid-phase process. As their names indicate, the chief difference is that in one case a liquid butane and in the other case, a butane vapor is passed, after drying and scrubbing, to the reactor or contactor where the butane is intimately contacted with a catalyst containing aluminum chloride. The activity of this catalyst is greatly increased by the addition of anhydrous hydrochloric acid.

Both types of processes were widely used during World War II, when the military demand for aviation gasoline and hence for alkylate, became enormous. After the war, operation of some of these plants was discontinued; others maintained operation. The design of postwar units were improved chiefly in the reactor section, with provision of extra reactors connected in series to improve the conversion per pass. For corrosion discussion purposes, however, the simplified flow diagram of a butane isomerization unit shown in Figure 1 will suffice to cover both processes. Typical operating conditions are shown in Table 1. It is not within the scope of this paper to compare the two processes.

Because of the nature of the catalyst and recycle of HCl gas, a corrosion tends to be related directly to the degree of aqueous hydrochloric acid formed from moisture pick-up, etc., and to sludges present in the system which absorb chlorides. A review will be made here of corrosion by hydrochloric acid followed by a discussion of the corrosion experiences in each of the processes, together with remedial steps.

Corrosion By Hydrochloric Acid

The rates of corrosion on carbon steel by dilute hydrochloric acid solutions are very severe indeed; for example, a metal loss of about 1 inch per year may be expected in the presence of about 2 percent hydrochloric acid at room temperature. HCl gas concentrations employed in isomerization processes are about 5-10

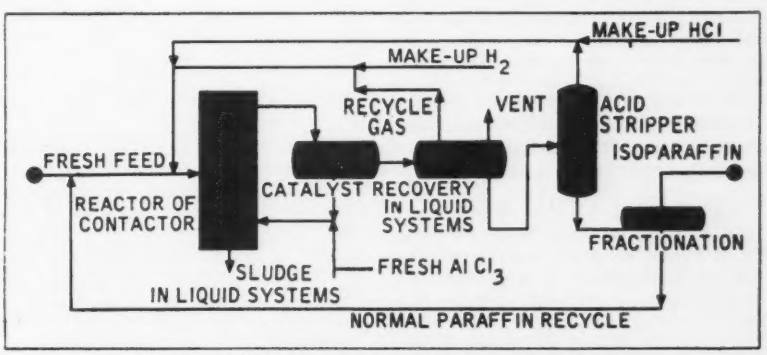

Figure 1—Typical aluminum chloride isomerization flow diagram.

★A paper presented at a meeting of the Western Region, National Association of Corrosion Engineers, Los Angeles, California, November 17-21, 1958.

(1) E. B. Backensto, Socony Mobil Oil Co., Inc., Paulsboro, N. J., Chairman.

(2) International Nickel Co., D and R Division, New York, N. Y.

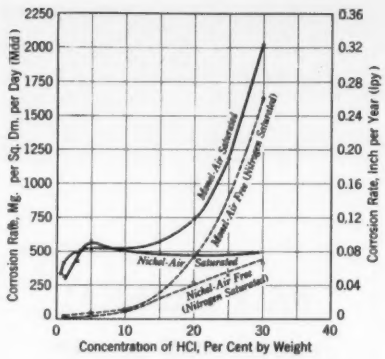
Corrosion Rate, Mg. per Sq. Dm. per Day (Mdd)
Corrosion Rate, inch per Year (ipy)
Monel-Air Saturated
Monel-Air Free (Nitrogen Saturated)
Nickel-Air Saturated
Nickel-Air Free (Nitrogen Saturated)
Concentration of HCl, Per Cent by Weight

Figure 2—Corrosion of nickel and Monel in hydrochloric acid at 30 C (86 F).

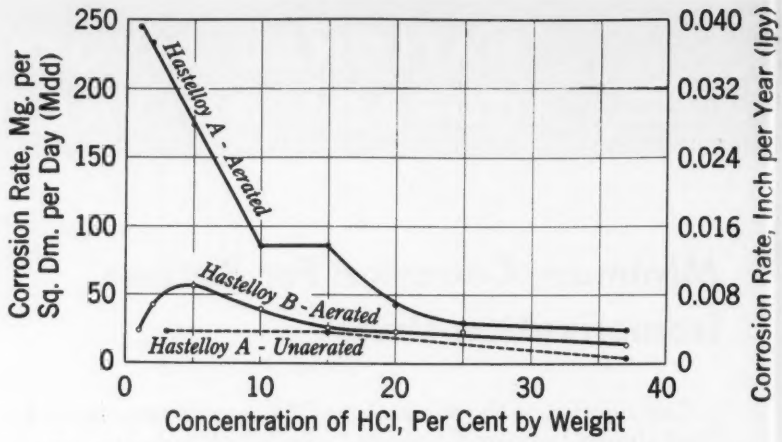
Corrosion Rate, Mg. per Sq. Dm. per Day (Mdd)
Corrosion Rate, Inch per Year (Ipy)
Hastelloy A - Aerated
Hastelloy B - Aerated
Hastelloy A - Unaerated
Concentration of HCl, Per Cent by Weight

Figure 3—Corrosion of Hastelloy alloys A and B in hydrochloric acid at room temperature.

percent which hydrolize in the presence of moisture to form dilute hydrochloric acid solutions.

The nickel and high nickel alloys are among the few metallic materials having useful resistance to hydrochloric acid solutions. Selection of the most useful and economical of these materials depends chiefly upon the concentration, aeration and temperature of the acid.

The performance of all of the high-nickel materials in hydrochloric acid solutions has been determined by service experience and by numerous laboratory and plant corrosion tests.

Acid Concentration

Figure 2 shows the effect of hydrochloric acid concentration upon the corrosion rates of Monel and nickel in air-free and air-saturated solutions at 30 C (86 F).

It will be noted that the corrosion rates of Monel and nickel at room temperature are approximately the same up to 10 percent acid concentration. In this concentration range, the corrosion rates of both materials in air-free solutions are normally under 0.01 ipy. The rates for both materials are increased considerably by saturating the solutions with air.

Above 15 percent acid concentration, the corrosion rates of Monel begin to increase rather sharply. In practice, applications of Monel at room temperature will be, in general, limited to concentrations under about 10 percent in aerated solutions and under about 20 percent in air-free solutions.

For handling hydrochloric acid in concentrations and temperatures above those indicated for the suitable performance of nickel and Monel, the most resistant of the high nickel alloys are the Hastelloy alloys A and B. Laboratory corrosion test data for both of these materials over the complete range of hydrochloric acid concentrations at room temperature, are presented in Figure 3.

Temperature Of The Solution

An increase in temperature increases the corrosion rates of both Monel and nickel in hydrochloric acid solutions, although nickel is affected more in this respect than Monel. Corrosion rates of Monel and nickel in 5 percent hydrochloric acid, both air-free and air-saturated, at temperatures up to 90 C (194 F) are shown in Figure 4. In unaerated 5 percent acid, Monel is usefully resistant up to about 75 C (167 F) and nickel up to 55 C (130 F). Applications of both materials, but particularly of Monel, in lower concentrations at higher temperatures are common. In most of the processes in which hydrochloric acid is formed as a result of hydrolysis of chlorides or chlorinated solvents, acid concentrations are less than 0.5 percent; corrosion from these acids is being withstood satisfactorily at temperatures of 300 to 400 F.

Hastelloy B is notable for its unusually high resistance to hydrochloric acid solutions of all concentrations and temperatures (see Figure 5). It is recommended particularly for service in hydrochloric acid solutions in the temperature range from 158 to 230 F and for handling wet hydrogen chloride gas. The penetration rate is only 148 mdd (0.023 ipy) in a 20 percent boiling solution, generally considered to be one of the most corrosive conditions.

In Figures 3 and 4, corrosion rates have been shown in air-saturated and air-free solutions in order to provide information as to upper and lower limits of corrosion which conceivably might be encountered in practice. In petroleum applications the system is not aerated and is usually free from oxidizing salts.

Experience with nickel and Monel equipment under various conditions of stress has demonstrated that these materials are not subject to stress-corrosion cracking in hydrochloric acid solutions.

The addition of nickel to iron increases its resistance to hydrochloric acid solutions at atmospheric temperature. The improvement is roughly in proportion to the nickel content where the nickel addition is above about 12 percent.

The nickel-iron alloys most commonly used with dilute hydrochloric acid at room temperature are the nickel and nickel-copper cast irons of the Ni-Resist series. Corrosion rates of Type 1 Ni-Resist (13.5 to 17.5 percent nickel, 5.5 to 7.5 percent copper, and 1.75 to 2.5 percent chromium) in unaerated hydrochloric acid solutions at room temperature are given in Table 2. Experience has shown that these corrosion rates can be applied equally as well to ductile Ni-Resist.

TABLE 1—Operating Conditions of Vapor Phase and Liquid Phase Process Units

Area of Unit	VAPOR PHASE PROCESS		LIQUID PHASE PROCESS	
	Temperature, °F	Pressure, psig	Temperature, °F	Pressure, psig
Driers	70	200	70	330
After Feed Preheaters	225	170	180	305
Reactors	225	160	195	300
Guard Chamber	225	150	180	170
Accumulator & Vent Gas Scrubber	50	125	100	...
HCl Stripper Column	220	280	...	325
Caustic Wash	100	...	140	...
Catalyst	Aluminum chloride		93% antimony trichloride & 7% aluminum chloride	
Average conversion per pass	35%–45%		50%–55%	

Vapor Phase Butane Isomerization

As pointed out in the previous section, the corrosive conditions result from formation of dilute HCl and sludge. The importance of keeping the system dry and free from sulfur, olefins and pentanes, cannot be over-emphasized. A number of case histories showing specifically the points where corrosion may be expected will be discussed.

The Bartlesville Unit of Phillips Petroleum Company was discussed recently in a paper by J. S. Connors and C. L. Seyer.[3] The unit was constructed of carbon steel with ⅛ inch corrosion allowance in the

HCl stripper, stripper feed tank and caustic wash tank. A gunite lining was used in the pretreater. Hastelloy B was employed for lining the reactor and for the piping from the pretreater to the reactors and from the reactors to the guard chambers. The remainder of the piping was steel and had a 3/16 inch to ½ inch corrosion allowance.

The most severe corrosion occurred in the carbon steel piping between the guard chamber and the effluent condenser, resulting in a fire. The average life of these steel pipes was found to be three years. At one time, Hastelloy B welded pipe was employed, but serious weld cracking and poor fusion in the longitudinal seams showed up.

The concrete linings in the pretreaters have required replacement at 4-year intervals, but have satisfactorily protected the vessels. The Hastelloy B lining in the reactors has required some maintenance, and has corroded at an average rate of .002 ipy. Glassed-steel vessels are being considered for replacement of the reactors. The seamless-steel tubes originally used in the effluent condensers have been replaced with Monel tubes, and the design of the condensers has been changed to utilize Monel-faced, double-tube sheets. The bottom portion of the stripper feed tank has been subjected to severe corrosion. This corrosion has been controlled by providing a ½ inch thick bottom lining and by sloping the vessel for positive drainage to external drain pots. The Hastelloy B lined valves have given satisfactory service, but have required repairs every three to four years. Solid Hastelloy B valves are now being used for replacement.

This experience contrasts with three units on the West Coast where carbon steel equipment was used throughout, with only moderate corrosion. Plant No. 1 showed mild attack on the reactors, guard chamber and around the HCl stripper but its equipment is still in repairable condition after 15 years of operation. Plants Nos. 2 and 3 also were installed during the war and are in even better condition. Plant No. 2 which has been in operation continuously, reported that the guard chamber needed relining. Loss of the ⅛ inch corrosion allowance corresponds to an average metal loss of .0089 ipy and this is the most severe corrosion.

The HCl stripper is corroding at the rate of about .0016 ipy. Some replacements have been required of bubble caps in the bottom part of this tower. Caps made of pressed steel did not stand up well, and cast iron bubble caps are now being used. The vent gas scrubber section experienced a corrosion rate of .003 ipy. Rates in the balance of this unit were negligible. Oil was used as a heat medium in the reactors and the porocell, saturated with aluminum chloride catalyst, was contained inside the tubes. No appreciable thinning of the tube ends could be noted.

The only alloy material being used in this plant is Hastelloy B for piston of a guard chamber bottom's reciprocating pump which was alternately subjected to the HCl-containing system and to the atmosphere which contained some moisture. Plant No. 3 came on stream in 1943 and was down from the period 1945 to 1954. Corrosion in this plant also has been negligible.

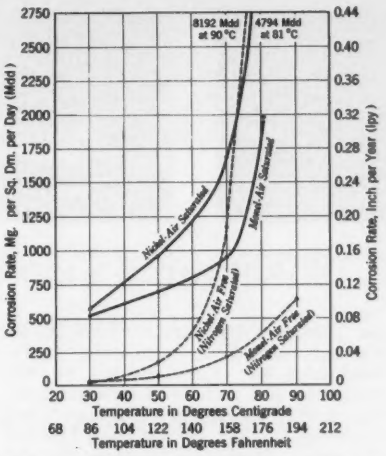
8192 Mdd at 90°C
4794 Mdd at 81°C
Nickel, Air Saturated
Monel, Air Saturated
Nickel, Air Free (Nitrogen Saturated)
Monel, Air Free (Nitrogen Saturated)
Corrosion Rate, Mg. per Sq. Dm. per Day (Mdd)
Corrosion Rate, Inch per Year (Ipy)
Temperature in Degrees Centigrade
Temperature in Degrees Fahrenheit

Figure 4—Effect of temperature on corrosion of nickel and Monel in 5 percent hydrochloric acid.

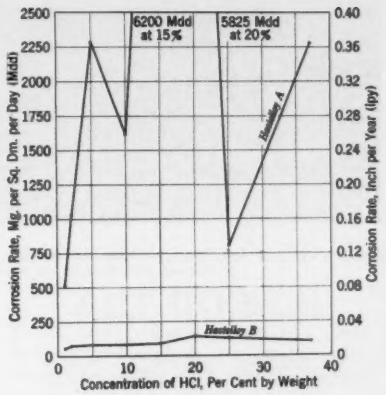
6200 Mdd at 15%
5825 Mdd at 20%
Hastelloy A
Hastelloy B
Corrosion Rate, Mg. per Sq. Dm. per Day (Mdd)
Corrosion Rate, Inch per Year (Ipy)
Concentration of HCl, Per Cent by Weight

Figure 5—Corrosion rates of Hastelloy alloys A and B in boiling hydrochloric acid (not aerated).

The secret of success lies in the careful control of feed stock quality. For example, total dissolved water in excess of 10 ppm was not tolerated. It usually ran in plant No. 3 at 1 to 3 ppm and at no time was free water present. Plant No. 2 reported that their feed stock was so dry that no drier was required. Also, keeping the pentane concentration below .01 percent and the olefin concentration below .005 percent was felt necessary. This was accomplished by acid treating of the feed which incidentally, also reduced the sulfur concentration to below 10 ppm. The feed was then sent to a butane splitter to remove the isobutane and light fractions. The bottom was rerun to remove pentanes. This way, essentially a pure n-butane feed stock was obtained. Formation of corrosive sludges in the system were for all practical purposes eliminated.

Plant No. 3 is very emphatic on control of water content. A Beckman continuous recorder was installed there on the feed stream. In addition, 18 analyses were carried out per day for water content on samples drawn from different locations.

Similar extreme precautions are taken when starting up the unit after shutdown to ensure removal of all moisture. Plant No. 2 circulates hot butane through the system until the unit is warm and dry, while plant No. 3 passes hot fuel gases until no moisture is found in the exit gases. After a prolonged shutdown, the steel is pickled to remove iron oxide scale before starting up.

It is felt by the operators that these precautions are a necessary part in the efficient operation of this type of unit. Keeping the system dry and keeping contamination out are the controlling factors in minimizing corrosion, rather than adjusting variables such as reactor temperature, pressure, HCl recycles, catalyst consumption, space velocity and conversion ratios.

TABLE 2—Corrosion of Type 1 Ni-Resist in Unaerated Hydrochloric Acid Solutions at Room Temperature

Acid Concentration, % HCl by wt.	Corrosion Rate, ipy	
	Type 1 Ni-Resist	Plain Cast Iron
1.8	0.005	0.9
3.6	0.015	..
5.0	0.018	1.5
10.0	0.016	1.2
20.0	0.045	..

Liquid Phase Butane Isomerization

A liquid butane is passed to the reactor, where it is contacted with a liquid catalyst consisting of a solution of aluminum chloride in antimony trichloride. Anhydrous HCl is added to the mixture as a promotor. The reactor is a vertical autoclave equipped with a mixing device to provide intimate contacting of catalyst and hydrocarbon.

Because of the corrosive nature of the catalysts, the reactors, vessels, pumps and transfer lines in catalyst service were clad or lined with nickel or Hastelloy B. The latter was preferred in piping, valves and pump construction. All other equipment was carbon steel.

This process did not suffer deactivation of catalyst to the same degree as the vapor phase process when olefins, pentanes and sulfur compounds were present. Catalyst activity was maintained constant through the continuous removal of degradation products resulting from reaction of impurities with the catalyst. This was achieved by means of the catalyst scrubber. The insoluble aluminum chloride-hydrocarbon complex sludge was rejected periodically from the bottom of the scrubber column.

Shell Development representatives recommend the following maximum impurity concentrations for their process: olefins .05 percent, pentanes 1.0 percent, sulfur .005 percent and water 10 ppm. Even though it is possible to operate plants with an even higher level of impurity without interference with the effectiveness of the process, catalyst consumption and sludge formation will in-

Figure 6—Reactor strip lining of Hastelloy B. Note severe stress corrosion in stressed area next to the welds.

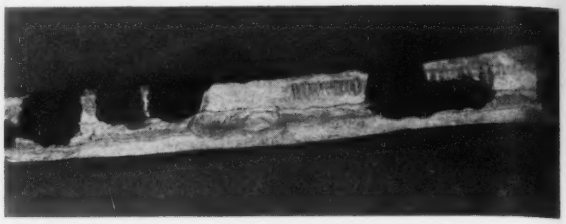

Figure 7—Dye checking of strip lining reveals cracks that showed up after a year's operation.

crease. The latter certainly plays a role in the extent of corrosion being experienced with these units. Here also, feedstock quality is a very important factor.

Several plants which have been operating this type of unit since 1943 have reported that in their twice-yearly shutdowns extensive repairs almost always had to be made. The reactor and catalyst removal column or guard chamber were the chief trouble spots.

Plant No. 1 reported about 2½ to 3 years life when using ⅛ inch nickel-lining in reactors. This recently was replaced with ¼ inch nickel-clad on steel. Besides a thinning of the nickel lining, troubles were experienced in the weld zones because of porosity and iron-dilution of the nickel welds. Plant four which had similar problems solved this by applying 2 inch-wide strips of nickel over the weld zones; another solution was the use of Hastelloy W welding rod. The present practice of heliarc welding reduces iron dilution substantially and has largely overcome this problem.

Lining of the reactor with Hastelloy B strips has been an alternate solution. This alloy, as pointed out before, has almost negligible corrosion rates in this system. Plant No. 1 which used Hastelloy B strip lining in one reactor, experienced severe stress corrosion in the stressed areas adjacent to the welds. Figure 6 is a photograph of a section of the failed strip lining showing the transverse cracking at the points where the Hastelloy B was preformed for making the overlap joint. Figure 7 shows the same section which was dye-checked from the back side. The cracks showed up after one year of operations, and became progressively worse with time. When overlaying these areas in repair, new cracks formed.

Hastelloy B work hardened very easily and evidently was sensitive to stress cracking in this service. The cracks occurred first in the bottom shell in the hotter part of the vessel. In one instance the impression of a hammer blow on a plate could be clearly distinguished during the first shutdown, and fine stress cracks noted at the periphery of the indentation. Iron dilution in the weld zones also was a problem here.

Plant No. 1, to overcome this problem, used a lapjoint when the previously Hastelloy B-lined vessel needed relining after three years of operation. The strips were preformed with a small "break." This practice eliminated corrosion in the weld zone, but stress cracking occurred in the forming zones. Bad undercutting at the weld zones was often the result. Several companies feel that the economical solution lies in the use of an integral-clad Hastelloy B vessel, say by using Lukens-clad or the Horton-clad process. For the nozzles solid Hastelloy B is suggested.

Severe attack also has been experienced on Hastelloy B agitators in cases where a lined agitator or cast vanes welded to a shaft were employed. Cast Hastelloy B seemed to have undesirable porosity; bad undercutting occurred where the vanes were welded to the stem. It has been suggested that use be made of solid Hastelloy B agitators fabricated from wrought material. Some stress corrosion also has been noted on the shaft. A Stellited agitator shaft sleeve needed replacing every two years. It is hoped by this company that Hastelloy D will perform better than Hastelloy B because of higher hardness and wearability. No corrosion has been experienced in the 30-inch manhole through which the agitator enters the reactor. This is attributed to the use of a butane flush system to continuously purge the packing glands and keep the HCl away from this area. This same principle of butane flushing is being applied to purge the packing glands of pumps.

The catalyst removal column or guard chamber also experienced corrosion. Five percent nickel-clad steel was used in the lower half up to between 3 and 4; solid nickel nozzles also were employed. Hastelloy B tubes were used in the internal reboiler which operates at 200 F and 40 psi steam was used as heat medium. Porosity and iron dilution at the nickel welds were the chief problems; relining after six years was necessary. The original Hastelloy B reboiler tubes are still in use and in good condition. The guard chamber uses carbon steel trays and cast iron bubble caps. Their life is estimated at five years by plant No. 1; the steel trays are being replaced with solid nickel trays.

The aluminum chloride saturators were made of carbon steel with Monel screens. The screens lasted about three years. The original carbon steel catalyst storage vessel and antimony trichloride melter were still in service after 15 years.

Plant No. 1 used cast Hastelloy B transfer lines in catalyst service. Replacement was required after six years not because of thinning of the wall but because of leaks through the casting. Now, wrought Hastelloy B piping is employed, a material which is felt to be much better, but which may be subject to troubles in the weld zones. Solid Hastelloy B valves are used in the catalyst system; carbon steel valves with Hastelloy B trim are used elsewhere. Carbon steel pipes can be used in other parts of the unit and a corrosion allowance is being applied. For safety reasons a number of tell-tale holes can be drilled to a depth equal to the metal thickness required for strength, leaving the corrosion allowances undrilled.

Conclusion

Irrespective of what process is being used, it seems extremely desirable to the authors to take all possible precautions to ensure that the feed is dry and contains a minimum of impurities such as sulfur, pentanes and olefins. Adequate guard chamber capacity is a must. The vapor-phase process shows very well how, when insisting on feed quality, corrosion can be kept at a very low and tolerable level. This is the first step applied in preventing corrosion problems; alloys can be used in this process in the critical locations when experience indicates their necessity. In the liquid-phase butane isomerization process, of course, alloys must be employed in the catalyst handling section. Hastelloy B is outstanding and lasts indefinitely if parts are not stressed. Otherwise nickel seems the more economical selection. In the most severe application (i.e., the reactors), 5 to 6 years life can be expected when using ¼-inch nickel clad steel or nickel-lining using the heliarc process to minimize iron dilution.

Acknowledgment

The writers wish to acknowledge the cooperation of companies such as Phillips Petroleum Company, Tidewater Oil Company, Shell Oil Company, Richfield Oil Corporation, and Union Oil Company of California. Their willingness in freely discussing problems experienced, is highly commendable. It is hoped that this article will meet its intended purpose, that is, to help those companies with butane isomerization units in the design or planning stage.

Reference

1. J. S. Connors and C. L. Seyer. Solutions to Corrosion Problems in Light-Hydrocarbon Liquid Plants. A paper presented before the API Division of Refining, Los Angeles, May 12, 1958.

NATIONAL ASSOCIATION of CORROSION ENGINEERS

Corrosion of Refinery Equipment By Aqueous Hydrogen Sulfide*

NACE TECHNICAL COMMITTEE REPORT
Publication 59–12

A Contribution to the Work of Group Committee T-8[1] On Refining Industry Corrosion, by Roy V. Comeaux[2]

Abstract

H_2S corrosion problems at temperatures below 400 F in the presence of water are presented for a wide range of refinery equipment. The most commonly accepted solutions to the problems of corrosion and fouling are the use of resistant alloys and/or neutralization plus film-forming corrosion inhibitors. Experience at several units employing steel condenser tubes exposed to waters contaminated with H_2S and other acids will be discussed. A theoretical study of the corrosion of steel in H_2S solutions reveals that prevention of high velocity, low pH exposures should be an effective control measure. Analysis of several failures of steel-tubed exchangers are discussed in the light of additional improvements that may be possible in design and selection of materials. Preliminary tests indicate that zinc or aluminum coated steel tubes may be useful in this environment.
3.4.8

Introduction

CRUDE OILS are generally contaminated by hydrogen sulfide, salts including magnesium chloride, organic compounds of nitrogen and sulfur of high molecular weight, and by acids generally referred to as naphthenic acids. This paper is concerned only with the corrosion problems experienced at temperatures below 400 F when handling hydrocarbons which contain sufficient water to produce aqueous phases at metal surfaces.

Due to the presence of hydrogen sulfide in the hydrocarbon and water phases and to the low solubility of 2-valent metal sulfides, sulfide scale or colloidal sulfides in process waters are commonly experienced. Detailed explanation of the other contaminants which influence metal corrosion rates is beyond the scope of this paper.

This paper discusses recent developments which encourage the use of mild carbon steel in light naphtha processing equipment under conditions which formerly were considered to be the province of alloys. A theoretical basis for these developments is presented. Crudes are first fractionated in pipe stills into fractions such as light crude naphtha, gas oils, and residuum stocks. The light naphthas are contaminated by the free hydrogen sulfide originally present in the crude and by hydrochloric acid resulting from the hydrolysis of magnesium chloride. To reduce corrosion of the condensing equipment, steel piping and pumps, ammonia is added equivalent to the hydrochloric acid present. Although the corrosion rates of steel in the aqueous systems containing ammonium chloride, some ammonium bisulfide and free H_2S are relatively slight compared to those obtained in the hydrochloric acid solutions, there is a steady build-up of sulfide scales which contribute to fouling of downstream equipment. Monel and Admiralty have been used widely in overhead condensing service.

Refinery equipment exposed to aqueous H_2S corrosion is shown in Figure 1.

Light crude naphtha is debutanized and then desulfurized in catalytic units to convert organic sulfides into hydrogen sulfide which can be removed by distillation. Throughout the sequence of processing steps, corrosion and fouling problems are minimized by application of ammonia, water and film forming organic inhibitors.[1]

The intermediate products from the pipe stills are cracked into gasoline boiling range components. The high molecular weight sulfur compounds decompose to hydrogen sulfide, and the nitrogen compounds decompose to compounds such as cyanogen and ammonia. The presence of cyanides in appreciable quantities promotes hydrogen blistering of steel. The harmful effects of cyanides can be reduced by air injection,[2] by water injection and by recycling under the proper conditions. Because of the latter and the small amount of water involved, injecting water soluble, filming inhibitors at a single point to protect several banks of exchangers from sulfide corrosion and fouling is relatively inexpensive.

Light hydrocarbons produced in distillation of crudes and cracking of intermediate distillates are contaminated heavily with acidic components. These streams are economically neutralized by Girbotol extraction of H_2S and CO_2, using water soluble amines which can be regenerated by heating and the addition of sufficient strong base to liberate the amine from its salts of the strong acids (thiocyanic, formic, acetic) and recycled to the absorbers for reuse.[3] This operation is included because it represents a corrective measure from the corrosion standpoint while simultaneously presenting a potentially serious problem in view of the possibilities for concentration of corrosives. The unit produces a gas stream containing 88 percent H_2S and 12 percent CO_2.

Corrective Measures

At the Baytown refinery, until recently it was necessary to use alloys in all water cooling and condensing equipment. In 1953, however, a combination of credits justified the adoption of a refinery-wide program of treating circulating waters to inhibit oxygen corrosion of metals and calcium compound deposition on hot surfaces. Success with this program now permits the use of mild carbon steel unless limited by oil-side corrosion. A better understanding of the nature of corrosion of steel in aqueous solutions containing hydrogen sulfide will reduce the frequency of rejecting steel from consideration.

Corrective measures vary according to specific economic factors at sites in the refining industry. It may be generalized, however, that neutralization, the use of alloys, partial removal of the contaminants from the feed, and film-forming inhibitors were introduced in the order shown. Inhibitor costs are about 0.1 cent per barrel of overhead naphthas.

Although the general level of success has been high, as indicated by a run length exceeding two years on a pipe still processing sour crude, equipment

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★ A paper presented at a meeting of the South Central Region, National Association of Corrosion Engineers, New Orleans, October 20-24, 1958.

(1) E. B. Backensto, chairman, Socony Mobil Oil Co., Inc., Paulsboro, N. J.

(2) Humble Oil & Refining Co., Baytown, Texas.

(3) US 2,718,454, Roger Wylie, issued Sept. 20, 1955.

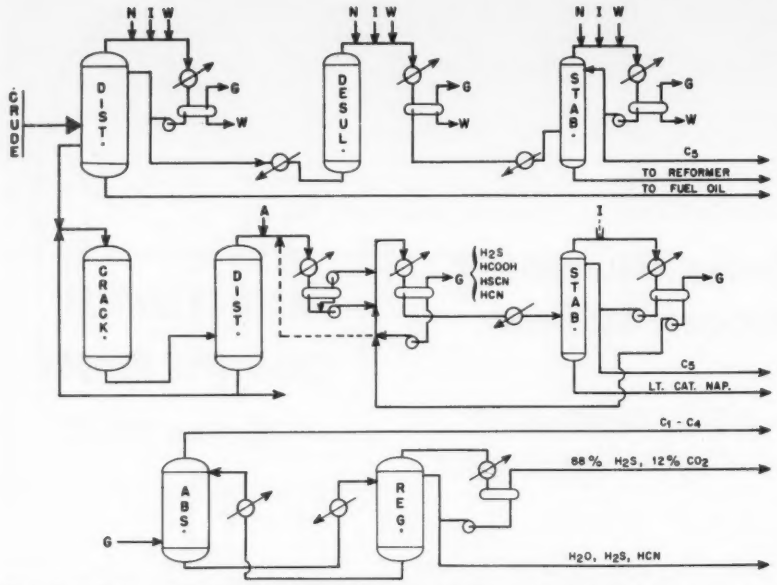

Figure 1—Refinery equipment exposed to aqueous H_2S corrosion. Code to letters is as follows: A—air, N—ammonia, I—inhibitor, W—water, G—gases.

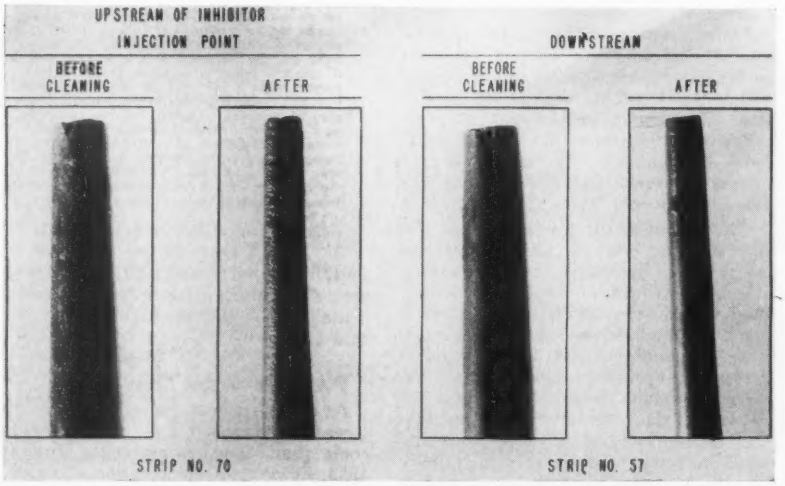

Figure 2—Inhibition of low temperature H_2S corrosion in pipe still. Coupon exposure was from 7-24-56 to 11-21-56. Strip No. 70 had a weight loss of 1.554 and corrosion rate of 11.68 mpy. Strip No. 57 had a weight loss of 0.046 grams and a corrosion rate of 0.35 mpy—a reduction of 97.2 percent in the corrosion rate.

failure analyses suggest that further improvements in service factor or reductions in investment and operating costs are possible and worth investigating. These analyses focus attention on the necessity for controlling mineral acidity by neutralization, the control point depending on metals of construction,[1] and for maintaining a high service factor on equipment installed for corrosion protection. In the critical overhead condensing, service failures caused by general corrosion are rare. Failures due to impingement, high velocity acidic washing, bimetallic corrosion, and high concentrations of complexing agents are common.

Solutions should be sought in the direction of minimizing the entry of materials which result ultimately in highly localized acidic conditions and in designing the equipment so that high velocity shortcuts can be avoided. A review of exchanger design practices appears to be in order. As will be shown, design should minimize velocities to permit the development of concentration polarization in the annular space between tubes and baffles. Tube composition and production practices affect service life. Continued improvement, possibly through tighter specifications, are to be expected.

Plant Results with Corrosion Inhibitors

Results with Inhibitor in Sour Naphtha Lines

The Baytown refinery crude distillation area is 1.7 miles pipeline distance from the rerun area. With the advent of desulfurization, caustic washing was discontinued. In anticipation of sulfide scale fouling of preheat exchangers on the desulfurization unit, inhibition of the naphtha lines was initiated. Effectiveness of one quart of inhibitor per thousand barrels was determined by extractable coupons. Representative results are presented on Figure 2 for a line handling sour naphtha about half of the time. Although the 11.68 mpy corrosion rate is not serious from service life of lines, it is serious from the standpoint of fouling of process equipment. For example: at a density of 125 pounds per cubic foot for sulfide scale, a corrosion rate of 1 mpy produces one cubic foot of scale per year per 1000 feet of 8-inch pipe. The inhibitor-corrosion product film on the downstream coupon before cleaning is noticeable.

TABLE 1—Corrosion Rates (mpy) on a Debutanizer Column Operated on Inhibited Feed for Six Months

Cold Feed	230 F Hot Feed	350–360 F Hot Bottoms	Cold Product
1.3.....	1.2–2.7	0.4–2.1	0.2–0.7

TABLE 2—Corrosion Rates of Coupons in Catalytic Reforming Units

	No. of Coupons Showing Rates:		
	Below 5 mpy	5 to 10 mpy	Above 10 mpy
Before Inhibition	2	3	10
After Inhibition	32	5	8

Effect of Inhibitor on Debutanizers

After six months operation on inhibited feed, a debutanizer column was inspected. The tower, reboiler and bottoms cooler contained large accumulations of sulfide scale. It was immediately suspected that corrosion rates had accelerated. Corrosion rates (mils per year) were found, using extractable coupons, as shown by data given in Table 1.

The free H_2S content in the hot bottoms exposure is very low. Data showed the large amount of scale found in the unit was old scale removed from steel surfaces in the charge line and within the unit by the detergent action of the corrosion inhibitor.

Results with Inhibitor at Hydrofining (Desulfurizing) Units

Debutanized naphtha containing one quart of inhibitor per thousand barrels is desulfurized ahead of the crude naphtha rerun unit. All of the exchangers, condensers and coolers are carbon steel. Hairpin tubes are employed in the exchangers; this makes the cleaning process more difficult. The feed is heated above 400 F on the tube side of the preheat circuit. Visual inspections of the tubes indicate that sulfide fouling is limited to the hottest pass of the last exchanger in which the tube metal temperature exceeds 450 F. It is doubted that sulfide

TABLE 3—Contamination Analyses Over Several Months in Catalytic Light Ends Charged to Girbotol Unit

Accumulator	Organic Acids, %	Cyanides, ppm	Thiocyanates, ppm
Depentanizer, F-1	Up to 0.76	6–600	126–131
Prefractionator, C-1	Up to 0.50	244–1014	76–166
Depentanizer, F-2	Up to 0.62	740–1790	12– 41

TABLE 4—Corrosion Rates (mpy) of Steel Equipment in the Girbotol Unit

Exposure	Control Measures Suspended	Control Measures Lined Out
Lean Amine Line, 280°–300°F	633	0.3–4.0
Reactivator O.H. Condenser Outlet	181	0.4–1.2
Condensate Reflux	153	0.2–1.0

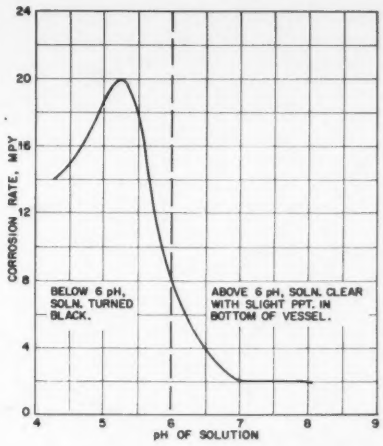

Figure 3—Effect of pH on corrosion rate of iron in solution of the same H_2S activity.

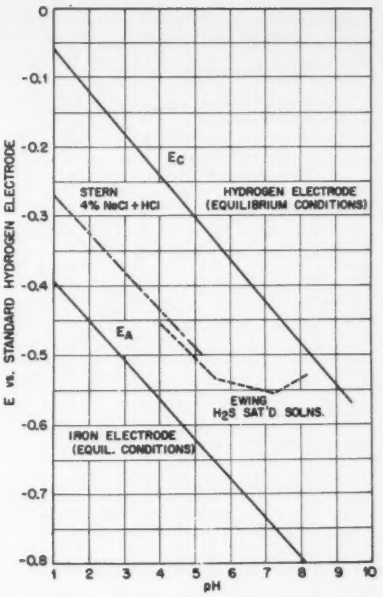

Figure 4—Effect of pH on potentials of iron electrodes.

corrosion above the 400 F level can be controlled with inhibitors of this type.

Application of corrosion inhibitors to protect steel in condensing service is not a panacea. In two cases the desired results were not obtained because of mechanical difficulties in controlling pH at the condensing surfaces and because of apparently high velocities through the annular spaces between tubes and baffles.

Results with Neutralization and Inhibition at Naphtha Rerun Units

Two identical systems of two columns each comprise the first step in feed preparation ahead of catalytic reforming units. The systems remove hydrogen sulfide by distillation. Inhibitor and ammonia are injected to overhead vapor streams; corrosion rates are monitored with extractable coupons. Results are generally excellent as shown by data given in Table 2.

Occasional high corrosion rates with inhibition are attributed to pH fluctuations experienced with manual control and to service factor problems on inhibitor pumps.

Results with Inhibitors in Amine Scrubbing Unit Handling Catalytic Light Ends

The extent of the contamination of the catalytic light ends charged to the Girbotol Unit is indicated by analyses of accumulator waters at random intervals over several months. The analyses are given in Table 3.

Presence of such contaminants in the Girbotol Unit charge streams results in gross contamination of the circulating amine and excessive corrosion of steel equipment if proper precautions are not taken. Corrosion rates (mpy) with and without control measures are indicated in Table 4.

Control measures, in addition to maintaining caustic alkalinity sufficient to neutralize strong acids in the feed, include continuous injection of a water soluble organic corrosion inhibitor to the reactivator overhead vapor line and continuous purge of a portion of the overhead condensate to discard cyanides. Low corrosion rates in reactivator overhead condenser outlet and in the condensate reflux were obtained in an aqueous phase saturated with H_2S and CO_2 under about two atmospheres of total pressure.[3] The present overhead condenser is stainless, but new units will be carbon steel.

Reactivator reboiler vapors contain little or none of the high boiling inhibitor or amine, and caustic alkalinity carryover is slight. A bypass condenser indicated these vapors containing H_2S and CO_2 to be corrosive to steel (40 to 80 mpy) but not to Type 304 (0.2 to 0.7 mpy).

Equations Expressing Electrochemical Reactions in Aqueous H_2S Corrosion of Steel

A promising approach to an improved understanding of the corrosion of iron in aqueous solutions containing H_2S was published by S. P. Ewing,[4] whose data are summarized in Figures 3 and 4. In Figure 4, data developed by Stern[5] for pure iron specimens in 4 percent NaCl solutions are included. Figure 3 shows the effect of pH on corrosion rate; Figure 4 shows the effect of pH on corrosion potential. Stern also developed the effect of pH on the cathodic polarization characteristics of pure iron; Figure 5 presents Stern's cathodic polarization diagrams for quiescent 4 percent NaCl solutions. Ewing's approach will be used in developing equations for corrosion currents in terms of pH values; Stern's results will be helpful in interpreting the equation.

Anode reaction-iron dissolution:

$$E_A = E^o_{Fe} + \frac{RT}{nF} \ln [Fe^{++}]_s \quad (1)$$

Cathode reaction-hydrogen evolution:

$$E_C = \frac{RT}{nF} \ln [H^+]_s - \frac{RT}{nF} \ln P_{H_2} \quad (2)$$

Where E_A and E_C are the reversible potentials of the iron and hydrogen electrodes respectively, $[Fe^{++}]_s$ and $[H^+]_s$ are the ion activities in the solution and at the electrode surfaces, P_{H_2} is in atmospheres, $\frac{RT}{F} = 0.0592$ at 25 C, and n is the number of electrons involved in the half-cell reaction.

Cell reaction and polarization:

Iron dissolution at the anodes (oxidation) in this case is associated with hydrogen evolution (reduction) at the cathodes. Both half-cell reactions are necessary to the corrosion process. The oxidation and reduction processes, however, result in appreciable changes in the compositions of the electrolytes contacting the anodic and cathodic areas with the result that the iron ion activity at the anode surface will become much larger than that in the solution, and the hydrogen ion activity at the cathode will become much smaller than that in the solution if polarization is allowed. It becomes necessary to consider three pH values (pH_{anode}, $pH_{solution}$, and $pH_{cathode}$), three iron ion activities, etc.

Polarization characteristics of metals are obtained by measuring the potentials at metal surfaces with respect to suitable reference electrodes as the current density collected or discharged by the surface is varied. Stern's cathodic polarization curves (Figure 5) for pure iron in quiet 4 percent NaCl solutions of various pH values are helpful in presenting this subject. It should be pointed out that corrosion rate (mpy) is linearly related to current density (i.e., micro-amperes per square centimeter). A number of investigators have presented evidence which permits a breakdown of the several polarization components, as shown in Figure 6.

In the center of Figure 6 the corrosion potential, $E_{Corrosion}$, and the corrosion current, $i_{Corrosion}$, are indicated for a

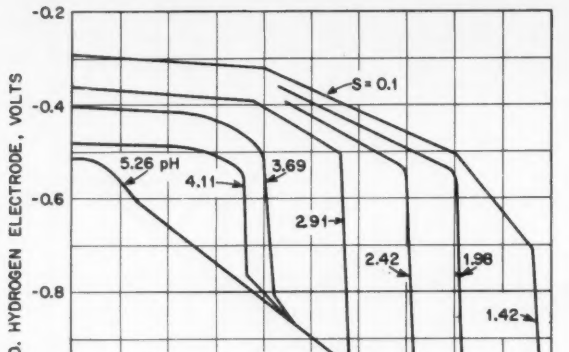

Figure 5—Effect of solution pH on cathodic polarization of pure iron in deaerated 4 percent NaCl in quiescent condition.

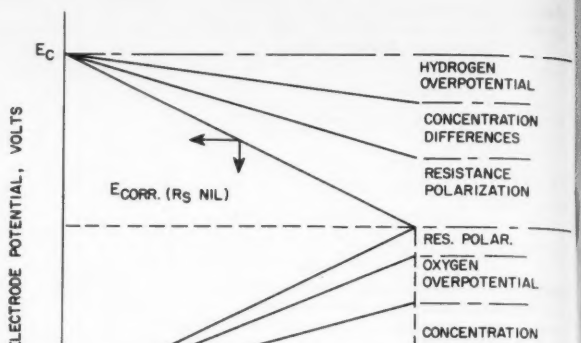

Figure 6—Polarization diagram

system having a negligible IR drop through the water phase. If the resistivity of the solution is high or the solution path is long, there may be an appreciable IR drop in the solution resulting in the corrosion potential being closer to the reversible potential of the cathode.

This assumes that corroding iron may be considered as a cathodically polarized hydrogen electrode with its own particular hydrogen over voltage constants. Stern[5,6] studied the corrosion of pure iron and the effects of alloying elements (copper, silicon, phosphorous, sulfur and manganese) on the corrosion of pure iron in acid solutions and deduced the following equation for situations in which cathode polarization is due solely to activation overpotential:

$$\text{Log } i_{\text{Corrosion}} = \text{Log } i_o - \frac{E_{\text{Corrosion}}}{b}$$

where b is the slope of the hydrogen overpotential line and i_o is the exchange current in amperes cm², a factor depending on the composition of the metal and on the nature of the corrosive solution. Carbon, sulfur and phosphorous accelerate the corrosion of iron in acids; copper and manganese reduce the deleterious effect to some extent.

Polarization, if permitted to occur, will decrease the rate of corrosion.

From Equations (1) and (2) and from Figure 6, the so-called "driving voltage" of the cell based on solution composition, $E_C - E_A$, can be equated to a number of terms dependent on concentration gradients in the electrode films, ohmic voltage drops and a term expressing hydrogen overpotential:

$$E_C - E_A = \frac{RT}{nF} \ln [H^+]_S - \frac{RT}{nF} \ln P_{H_2} - E^\circ_{Fe} - \frac{RT}{nF} \ln [Fe^{++}]_S \quad (3)$$

$$E_C - E_A = \frac{RT}{nF} \ln \frac{[Fe^{++}]_A}{[Fe^{++}]_S} + i_A \rho_A l_A + i_C \rho_C l_C + \frac{RT}{nF} (pH_C - pH_S) + b \log \frac{i_C}{i_o} + i_S \rho_S l_S \quad (4)$$

in which:

- i = current density, amperes per cm².
- ρ = resistivity, ohm-cm, a function of electrolyte concentration.
- l = thickness of film or length of electrolyte path, cm.
- b = slope of the hydrogen overpotential curve, which is a function of metal composition and environment.
- i_o = exchange current, which is also a function of metal composition and environment.

Subscripts A, C, and S refer to anode, cathode and solution values.

Equations (3) and (4) can be reduced to

$$2.303 \frac{RT}{2F} \log \frac{[H^+]_C^2}{P_{H_2}[Fe^{++}]_A} - E^\circ_{Fe} = b \log \frac{i_C}{i_o} + i_A \rho_A l_A + i_C \rho_C l_C + i_S s l_S \quad (5)$$

It is desired to introduce the hydrogen sulfide variable into Equation (5). At a given temperature and pressure, activity of a dissolved substance in equilibrium with its solid phase is constant. In the case of iron sulfide:

$$[Fe^{++}] \cdot [S^=] = C_{Fe^{++}} \cdot f_{Fe^{++}} \times C_{S^=} \cdot f_{s^=} = K_s f^{\pm 2} \quad (6)$$

where [ ] denotes activities, mols per liter
C_{ion} denotes concentration, mols per liter
f_{ion} denotes activity coefficients, which vary with ionic strength and temperature
K_s denotes solubility product

As an approximation, it will be assumed that the activity product and the solubility product are synonymous though it must be remembered that an increase in the number of dissolved ions of any specie will actually increase the solubility of iron sulfide. Therefore,

$$[Fe^{++}]_A \cdot [S^=]_A = 3.7 \times 10^{-19} \quad (7)$$

If the product becomes less than 3.7×10^{-19} due to a drop in pH, iron sulfide would dissolve and the cell is described by Equation (5). The product cannot exceed 3.7×10^{-19} without precipitation. By resorting to the dissociation constants for H_2S in aqueous solution ($K_1 = 10^{-7}$; $K_2 = 10^{-14}$) and Equation (7), the hydrogen sulfide variable and the hydrogen ion activity at the anode are introduced into Equation (5).

$$[S^=]_A = \frac{10^{-21} [H_2S]_A}{[H^+]^2_A} \quad (8)$$

$$[Fe^{++}]_A = \frac{3.7 \times 10^{-19} \cdot [H^+]^2_A}{1.0 \times 10^{-21} \cdot [H_2S]_A} \quad (9)$$

$$2.303 \frac{RT}{2F} \log \frac{[H^+]^2_C \cdot [H_2S]_A}{P_{H_2} \cdot 370 [H^+]^2_A} - E^\circ_{Fe} = b \log \frac{i_C}{i_o} + i_A \rho_A l_A + i_C \rho_C l_C + i_S \rho_S l_S \quad (10)$$

Equation (10) applies only when conditions produce an iron sulfide film at the anodic area. Ewing discussed the importance of maintaining a high sulfide ion-to-iron ion ratio to assure precipitation at the metal surface and the preservation of a protective film of iron sulfide.

Sulfide ion activity is extremely sensitive to pH fluctuations, and the ratios are even more sensitive. Figure 7 presents the ratios for iron, zinc and copper sulfides. In the absence of iron sulfide precipitation at the anodic surface, Equation (5) would apply. When the latter prevails, the ratio of iron ions to sulfide ions is high and precipitation occurs in the solution. In systems where solution pH changes, the difference in $(pH_C - pH_A)$ will not be constant and corrosion rates will vary. It is not surprising that laminar non-protective sulfide scales are encountered.

Equation (10) can be simplified for a better appreciation of the factors that must be controlled to minimize corrosion of steel in aqueous H_2S exposure.

At 25 C, Equation (10) becomes

$$b \log \frac{i_C}{i_o} + i_A \rho_A l_A + i_C \rho_C l_C + i_S \rho_S l_S = 0.364 - 0.0592 (pH_C - pH_A) + 0.0296 \log [H_2S]_A - 0.0296 \log P_{H_2} \quad (11)$$

At the particular conditions of H_2S saturation at one atmosphere total pressure, $[H_2S]_A$ is approximately 0.1 mol per liter and P_{H_2} is 1.0. The last term drops out and the next last is about —0.0296. The equation further simplifies to

$$b \log \frac{i_C}{i_o} + i_A \rho_A l_A + i_C \rho_C l_C + i_S \rho_S l_S = 0.334 - 0.0592 (pH_C - pH_A) \quad (12)$$

From Equation (12), it is readily seen that the larger the difference in $(pH_C - pH_A)$, the smaller will be the corrosion currents. The difference will be largest when conditions permit concentration polarization at both the anodes and cathodes. Concentration polarization cannot be obtained without finite values of l_A and l_C, such that larger values of l_A and l_C result in smaller values of i_A and i_C. Resistivities in many cases can be increased by using steam condensate as dilution water. Methods of controlling b and i_o are not so apparent; Stern[5, 6] has done outstanding work in this field. It is also readily seen that for the corrosion currents to approach zero, $(pH_C - pH_A)$ must approach 5.6, the horizontal separation in pH units between the hydrogen and iron electrode lines on Figure 4. In severely acid conditions (below 4 pH) and under conditions of high velocity, this pH difference can be approached only at excessively high corrosion rates.

It may be concluded that successful use of steel in aqueous, H_2S-contaminated systems depends on adequate pH levels and control and on design conditions for the various items of equipment which will allow concentration polarization to occur and inhibitor films to be effective. As an alternate to more exacting pH control, the use of coated steel tubes in condensing service is being explored.

With respect to design conditions, it can be deduced from the data in Figure 5 that a film at least 10^{-4} centimeters thick (one micron) and possibly 10^{-3} cm thick (10 microns) must be permitted to assure concentration polarization of the cathodes. From a control standpoint, pH must be maintained high enough so this polarization will occur at a low corrosion rate.

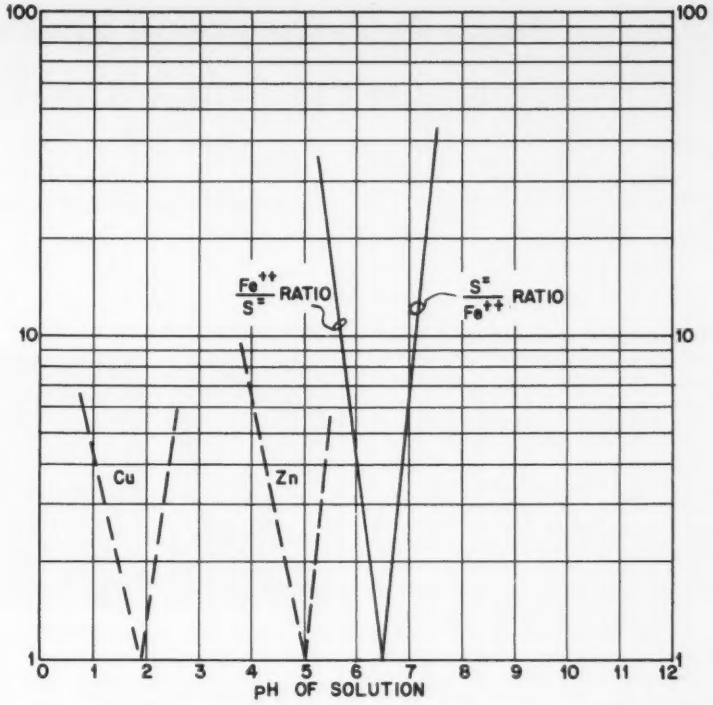

Figure 7—Comparison of pH values above which sulfide ion activity exceeds metal ion activity.

Current density, microamperes per cm^2, can be expressed in terms of the number of hydrogen ions reduced at the cathode per second per cm^2. ($1\ \mu$ Amp/$cm^2 = 6.24 \times 10^{12}$ H^+/sec.-cm^2). At any given pH, the thickness of a column 1 cm^2 containing this number of hydrogen ions or a multiple thereof can be calculated.

Exploratory Tests Based on Theory

The usefulness of steel tubes in H_2S contaminated systems requires that pH values be substantially neutral or that inhibitors be employed and pH maintained above about 6.5. A similar consideration of solubility product values for zinc sulfide indicates a pH of 5.0 would be permissible. A laboratory test of zinc metal in 1000 ppm NH_4Cl saturated with H_2S by bubbling at a pH of 4.0 produced a corrosion rate of 0.2 mpy. The specimen was covered with an even, finely divided film of white zinc sulfide. In concentrated ammonium hydrosulfide, however, galvanized steel corrodes with vigorous hydrogen evolution due to zinc-ammonia complex formation until all of the coating dissolves. The concentrations of complexing agents which can be tolerated have not been estimated.

Aluminum coated specimens resisted this corrosive, with corrosion rates of less than 1 mpy, with some evidence of localized attack where iron sulfide deposited on the aluminum coating.

Use of zinc coated steel has a decided advantage in view of its high hydrogen overvoltage compared to bare steel. Galvanized coupons have been obtained for testing in process equipment.

Conclusions

An improved understanding of the measures that should be taken to minimize corrosion of steel surfaces exposed to water solutions containing hydrogen sulfide and other contaminants results from a study of corrosion reaction electrochemistry. An expression can be developed which easily demonstrates the beneficial results of certain design and operating practices which are controllable. Plant experience has demonstrated that additional attention should be directed to condenser design to assure effective concentration polarization at metal surfaces. Exploratory tests indicate that galvanized steel might be useful in H_2S contaminated water containing minor amounts of complexing agents.

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Some Experiments on Internal Oxidation Of Nickel Chromium Alloys*

By H. R. COPSON and F. S. LANG

Abstract

A rare but deep internal oxidation of nickel-chromium alloys sometimes occurs in service. On some occasions oxidation is accompanied by deeper carburization or sulfidation, but in other cases these features seem absent. Laboratory tests showed that, with less than 9 percent chromium, exposure to air produced a thick external oxide under which there was some internal oxidation. With more chromium, however, attack in air was at a low rate. Several unsuccessful attempts to produce internal attack in high chromium alloys are described briefly. Exposure to carbon monoxide at 1750 F was found to cause rapid simultaneous carburization and internal oxidation of some nickel-chromium alloys. The addition of iron and silicon to the alloys slowed the attack. The addition of 2 percent columbium or 2 percent titanium prevented the attack completely. Some comments are made about mechanism. 6.3.10

NICKEL-CHROMIUM alloys sometimes undergo an internal type of oxidation in service. The chromium is oxidized at depth, and the remaining metal is strongly magnetic. The alloy becomes brittle and can be broken easily, showing a dark green fracture. After polishing, however, it looks bright and metallic. Micro-examination shows subsurface oxidation with oxides at the grain boundaries and within the grains. The attack may be uniform or it may occur locally in the form of blisters. The surface swells and cracks. This type of attack is observed in atmospheres which may be characterized as marginally oxidizing, that is, as oxidizing to chromium and reducing to nickel. The attack is observed usually in the temperature range from 1500 to 1800 F, but this may not be significant because attack has been observed at temperature as low as 1200 F and as high as 2100 F. The rate of attack varies considerably and may range from 0.04 to 0.7 ipy over extended service periods.

This internal oxidation has been difficult to reproduce for study in the laboratory. Bucknall and Price[1] reproduced the attack in a marginal atmosphere by periodic injection of an oil which caused carburization. They regarded all attack of this type as being preceded by carburization, and oxygen as diffusing inwards through the chromium depleted matrix and preferentially oxidizing the carbides. This explanation does not seem correct because severely carburized alloys do not oxidize at high rates.[2,3] Bucknall and Price noted that iron and silicon in the alloy were helpful, and suggested that these two materials scavenged oxygen from the surface layers, thus reducing the oxygen diffusion rates.

Dovey and Jenkins[4] concluded that a silica-rich film probably formed a barrier to carbon diffusion and that the silica may seal rifts in the normal oxide scale through which carbon would otherwise diffuse. The beneficial effect of iron was attributed to its greater solubility for carbon.

Some observed failures seemingly were not accompanied by carburization. ASTM Committee B-4, Sub-IX, developed a test procedure.[5] However, there has been little success in producing severe attack of high chromium alloys using this test procedure.[6]

Lang Copson

FRANCES S. LANG—With International Nickel Co., Inc., since 1951, she has a PhD in physical chemistry, New York University, 1952. She is research chemist, Corrosion Section, Research Laboratory, Bayonne, N. J.

HARRY R. COPSON—Head of the Corrosion Section, Research Laboratory, The International Nickel Co., Inc., Bayonne, N. J. he has been active in engineering associations for many years in organizing and managing corrosion control work. He is a member of many technical committees, has authored numerous papers and has a PhD from Yale University, 1932.

Spooner, Thomas and Tomassen[6,7] considered carburization irrelevant and described the basic reaction as internal oxidation at oxygen pressures low enough to leave the nickel intact and to oxidize the chromium. They observed rapid deterioration of 90-10 nickel chromium alloys at the bottom of long narrow thermo-couple tubes where the air was depleted and have reproduced the attack by placing 90-10 nickel chromium and nickel oxide in a vacuum and heating to 1820 F. The difficulty here is that similar attack seems not to have been produced on higher chromium alloys such as 80-20.

Also, oxidation resistance of nickel-chromium alloys has been reported increased by exposure to marginally oxidizing conditions which promote the formation of chromic oxide.[8]

The nature of the protective oxide coating on the surface of the metal is important. Normally a protective oxide forms on high chromium alloys, but, when internal oxidation occurs, this protection apparently breaks down. Undoubtedly an important consideration is the relative diffusion of chromium and oxygen (and carbon and sulfur) within the alloy.

A Typical Example

A typical example of this internal oxidation illustrated in Figure 1 shows the appearance after two years in a controlled atmosphere furnace of some 80-20 nickel-chromium hanger hooks used to support the heating elements. The furnace operated at 1650 to 1700 F with an atmosphere made by burning one part natural gas with seven and one-half parts of air. The attack occurred where the hooks passed through slots in the brick roof, indicating that more air than present in the furnace was needed to support the attack.

As can be seen in the figure, the attack was uneven. Unattacked areas retained their initial diameter and had a thin dark oxide coating. The attacked areas had increased in diameter and were a dull matte color. Where the growth was pronounced the oxide was cracked. All the fractured surfaces were a dark greenish color. The attacked areas were strongly magnetic.

Figure 2 shows a cross section through the hooks. In the attacked areas there was oxide at the grain boundaries and within the grains. Varying considerably from place to place, depth of penetration at any one place was uniform. This seems typical of sub-scale oxidation controlled by the inward penetration of oxygen. The attack was no deeper under the large cracks shown in Figure 1 than elsewhere. This indicates that the oxide formed was not much of a barrier to further oxidation. The metallic core in Figure 2 contains carbides, which were more massive than in the unattacked metal. Hence there was an accumulation of carbon ahead of the internal oxidation.

Results of chemical analyses made on different portions of the hooks are given in Table 1. The composition of the unaffected metal was typical of 80-20 nickel-

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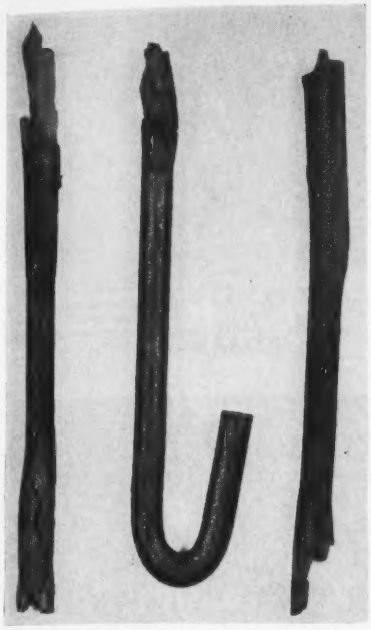

Figure 1—Local attack on some 80-20 nickel-chromium hanger hooks after 2 years exposure in a controlled atmospheric furnace.

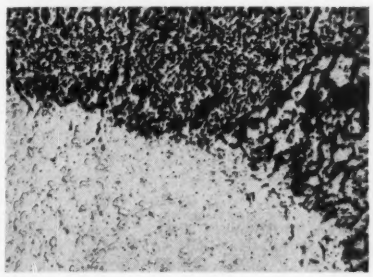

Figure 2—Cross section of nickel-chromium hanger hooks exposed 2 years in a controlled atmosphere furnace. Internal oxidation is shown at the top and carburization of the core of the hooks at the bottom. Unetched, 100X.

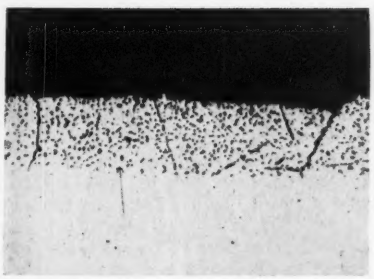

Figure 3—Cross section showing internal oxidation in an alloy containing 6 percent chromium and 94 percent nickel after 4 days in air at 1900 F. Unetched, 250X.

chromium heat resisting alloys, the balance unaccounted for in the table probably being mostly iron and manganese. The metallic core under the attacked areas was enriched in carbon although the 0.28 percent carbon present does not indicate severe carburization. If all the 0.07 percent carbon in a ¼-inch diameter rod were forced into a ⅛-inch diameter core, the carbon level would rise to 0.28 percent. The zone of internal oxidation, of course, contained oxygen. If all the chromium, iron, silicon and manganese are calculated as oxide, the total is still not 100 percent. About 7 percent of nickel has to be present as oxide also. On this basis some 63 percent of metallic nickel remained mixed with the oxides.

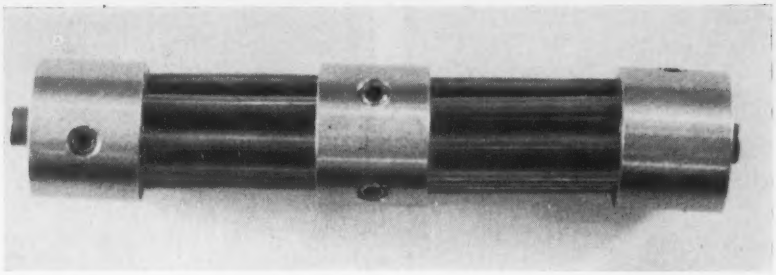

Figure 4—Holder for rod specimens.

TABLE 1—Chemical Analyses of Hanger Hooks Shown in Figures 1 and 2

	Unaffected Metal, %	Metallic Core Under Attacked Areas, %	Zone of Internal Oxidation, %
Nickel	78.09	76.93	70.00
Chromium	18.86	19.11	16.11
Silicon	1.12	1.13	1.08
Carbon	0.07	0.28	....

Examples of Internal Oxidation

The authors' laboratory has examined about 30 instances of rapid internal oxidation of nickel-chromium alloys during the past 15 years. Many of these involved carburization or sulfidation. Instances of internal oxidation apparently free of contaminating influences have also occurred. Curiously, in many installations where the conditions seemed identical with those where internal oxidation occurred, nickel-chromium alloys have withstood the attack. The attack that did occur in some instances needs explanation. In some cases an alloy more resistant to this type of attack could be substituted, but often there were good reasons for preferring a high nickel alloy.

Carburization and Oxidation

An instance of internal oxidation which was associated with carburization occurred in some 80-20 nickel-chromium heating elements which were used in a controlled atmosphere furnace (40 percent carbon dioxide, 11 percent carbon monoxide, 16 percent hydrogen, balance nitrogen) which operated at 2050 F. There was practically no attack on the heating ribbons at distances more than six feet from the furnace entrance. Here the dimensions of the ribbon were 1.5 by 0.09 inch in cross section.

Near the furnace entrance there was severe attack. Here the minimum thickness of the ribbon after six months of service had increased to 0.12 inch, and there remained only a small amount of the original metal. At the edges where there was no metallic core remaining, the oxide had split and curled outwards. The split was in the middle of both edges and about ¼-inch deep. The curling opened the mouth of the split a distance of about 0.1 inch. There were other cracks in the oxide elsewhere on the surface. These observations again indicate that the oxidation was occurring at the scale-metal interface and that extra air entering through the furnace door helped accelerate the attack.

Micro-examination of a cross section showed internal oxidation at the grain boundaries and within the grains. Carburization was present underneath the oxide. The degree of carburization paralleled the amount of oxidation, both decreasing with distance from the furnace entrance. Evidently the work was carrying some oil or carburizing material into the furnace. Because of the small amount of metallic core remaining in the most severely attacked areas, a quantitative analysis showing the maximum carburization could not be obtained, but it was quite severe.

In addition to 80-20 nickel-chromium heating elements, other instances of internal oxidation associated with carburization have been examined in the laboratory. One group of these involved Inconel carburizing boxes. Although carburization had sometimes proceeded all the way through the wall thickness, internal oxidation was confined to the inside. There was never more than superficial oxidation of the carbides on the outside.

Another group of these boxes involved Inconel thermocouple tubes inserted into furnaces containing carburizing gases where the internal oxidation was confined to the outside of the tubes on the portion within the refractory wall. These conditions were similar to those of the hanger hooks already described although the carburization was more pronounced.

In some of these cases the carbides appear to be attacked directly and converted to oxides; at least the two substances appear in contact. In other cases there is what might be called a decarburized zone between the carbides and oxides.

Some cases of internal oxidation have occurred in marginally oxidizing gases which contained carburizing constituents but where no carburization of the Inconel was detected. These results may indicate that under proper conditions a small amount of carburization may trigger internal oxidation. In other cases the carburization was so extensive that analyses

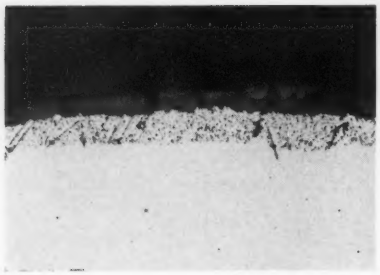

Figure 5—Cross section showing internal oxidation in an alloy containing 8 percent chromium and 92 percent nickel after 8 days in air at 1750 F. Unetched, 250X.

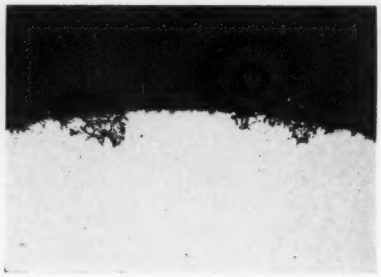

Figure 6—Cross section showing internal oxidation in an alloy containing 14 percent chromium and 86 percent nickel after 1 day in dry carbon monoxide at 1750 F. Unetched, 100X.

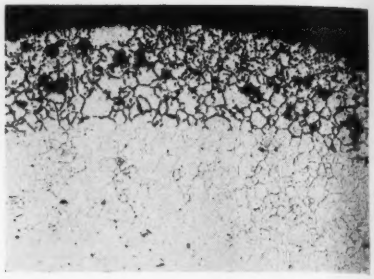

Figure 7—Cross section showing internal oxidation in an alloy containing 14 percent chromium and 86 percent nickel after 4 days in dry carbon monoxide at 1750 F. Unetched, 100X.

TABLE 2—Results of 4 Day Tests in Air At 1900 F*

MATERIAL		Gain in Wt., Mdd	Thickness of Oxide Layers, Mils		Magnetic Response at End of Test[b]
Ni %	Cr %		Continuous Outer Scale	Zone of Internal Oxidation	
100	0	570	5	0	Magnetic
97	3	870	5	3	Magnetic
94	6	690	10	3	Magnetic
91	9	310	6[a]	3[a]	Magnetic
91	9	56	7[a]	5[a]	Weakly-magnetic
88	12	...	Thin	Nil	Non-magnetic
85	15	24	Thin	Nil	Non-magnetic
82	18	20	Thin	Nil	Non-magnetic

* Specimens 0.375 x 1 x 0.03 inch in size.
[a] Spotty or irregular attack
[b] Alloys containing 6 percent or more of chromium were non-magnetic before exposure.

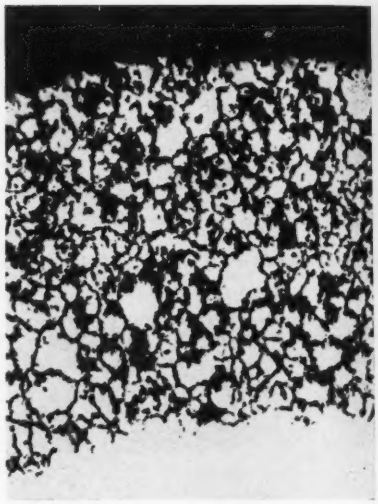

Figure 8—Cross section showing internal oxidation in an alloy containing 14 percent chromium and 86 percent nickel after 8 days in dry carbon monoxide at 1750 F. Unetched, 100X.

showed the presence of more than one percent carbon.

Sulfidation and Oxidation

An instance of internal oxidation associated with sulfidation occurred in some Inconel strip used to convey steel through an annealing furnace at 1800 F. The furnace gas averaged 8.6 percent carbon dioxide, 14 percent carbon monoxide, 10.5 percent hydrogen and the balance nitrogen. After three months of operation, attack was severe in some localized areas.

Micro-examination showed the typical internal oxidation had occurred and that the oxidized layer was underlain by a zone of sulfidation. Oil from the work probably caused the local sulfidation, and this led to the internal oxidation.

Several similar cases have been observed. This type of attack was rapid in some conveyer rods and bolts where they passed through openings in the furnace cover. The parallelism with internal oxidation associated with carburization is striking. The remedy is to eliminate the source of the sulfur contamination.

Internal Oxidation Without Contaminating Influences

The most puzzling instances of internal oxidation occur where there are no obvious contaminating influences. An instance of this occurred in an Inconel retort for making dissociated ammonia. The retort was heated electrically to 1750 to 1800 F. After one year of service, internal oxidation on the outside of the retort had almost penetrated the ¼-inch wall.

The retort's outer surface was magnetic and brittle. A few hammer blows would fracture the specimens, with the fractured surfaces having a typical dark green color. On a cut edge, the oxidized zone still appeared metallic.

Micro-examination showed uniform internal oxidation from the electrically heated side to a depth of 50 to 75 percent of the wall thickness with some fingers of oxidation extending deeper. On the ammonia side there was an altered layer about 0.010 to 0.015 inch deep which probably had been nitrided. In between these two zones of attack the Inconel appeared normal. No evidence of any carburization, sulfidation or other contamination was found.

In this and similar failures, weld metal resisted internal oxidation. The weld metal differed from the wrought Inconel in composition by containing some columbium.

Other instances of internal oxidation have occurred on one side of an Inconel component where the other side was exposed to gas containing hydrogen. In addition to attack on the outside of Inconel equipment containing dissociated ammonia, subscale formation has occurred on the inside of Inconel thermocouple tubes exposed to marginally oxidizing gases containing hydrogen on the outside. Presumably there was nothing but air (or oxygen depleted air) on the inside of these tubes. Sometimes there was carburization on the outside of the tubes, but often this was shallow and remote from the internal oxidation. In one of these tubes internal oxidation was quite deep at the mid portions but non-existent at the closed end, indicating that real oxygen starvation stops the attack.

Laboratory Attempts to Produce Internal Oxidation

Several attempts were made to reproduce internal oxidation in the laboratory. These can be divided into four groups involving exposure to wet hydrogen (which was marginally oxidizing), to carburizing gases, to air and to carbon monoxide.

The tests in wet hydrogen were unsuccessful. These included steady exposure of Inconel to wet hydrogen for four days at 1400, 1600, 1800 and 2000 F and for 28 days at 1800 F. The moisture content of the hydrogen had no great effect nor did prior oxidation in air. Cyclic exposure to air and to wet hydrogen was tried without much luck although there were occasional small areas of incipient attack.

Cyclic exposure to carburizing conditions and to wet hydrogen likewise was unsuccessful. In some separate tests, Inconel was so severely carburized that it became magnetic, and portions were then exposed for four days to wet hydrogen at 1750 F and for four days to air at 1800 F without rapid attack. The tests in air and in carbon monoxide, however, did produce some significant results.

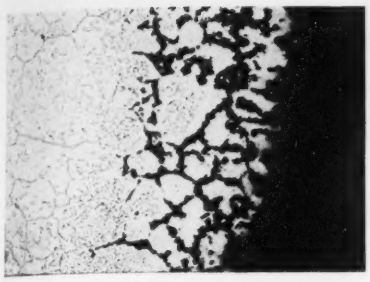

Figure 9—Cross section of alloy containing 14 percent chromium, 6 percent iron, 5 percent copper and balance nickel after 8 days in dry carbon monoxide at 1750 F. Carburization and oxidation are shown. Etched with electrolytic oxalic acid, 250X.

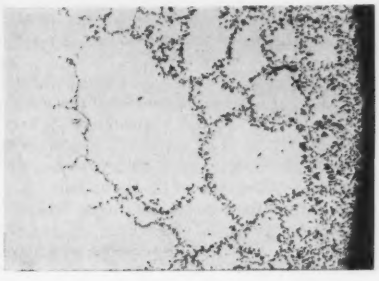

Figure 11—Cross section of alloy containing 8 percent chromium and balance nickel after 8 days in wet carbon monoxide at 1750 F. Unetched, 100X.

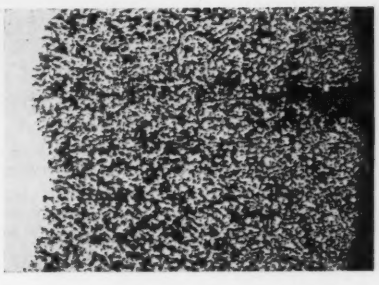

Figure 12—Cross section of alloy containing 20 percent chromium and balance nickel after 8 days in wet carbon monoxide at 1750 F. Unetched, 100X.

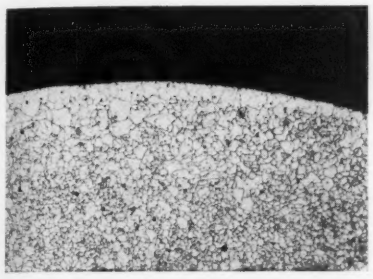

Figure 10—Cross section of alloy containing 16 percent chromium, 7 percent iron, 2 percent columbium and balance nickel after 8 days in dry carbon monoxide at 1750 F showing negligible attack. Etched with electrolytic oxalic acid, 100X.

TABLE 3—Results of 8 Day Tests at 1750 F[1]

COMPOSITION OF SPECIMENS				Gain in Wt., Mdd			Max. Penetration of Oxide from Surface in Mil		
Ni %	Cr %	Fe %	Other %	In Air	In CO	In Wet CO[2]	In Air	In CO	In Wet CO[2]
96	4	....	.....	52	210	100	2.2	13	41
92	8	....	.....	43	190	160	2.4	7	37
86	14	....	.....	3	460	340	<1	25	20
80	20	....	.....	1	300	470	<1	24[3]	32
80	14	6	.....	2	220	130	<1	9	6
66	14	20	.....	3	210	220	<1	9	12
46	14	40	.....	5	50	10	<1	14[3]	1
60	20	20	.....	5	40	40	<1	2	2[3]
Bal.	14	6	5.0 Cu	5	440	200	<1	10	9
Bal.	14	6	3.5 Mo	5	130	10	<1	5	5[3]
Bal.	14	6	2.0 Si	2	30	20	<1	5[3]	2[3]
Bal.	15.8	6.9	2.0 Cb	5	10	3	<1	0	0
Bal.	15.3	6.4	2.3 Ti and 0.7 Al	8	20	15	<1	2	1

[1] Specimens 0.1 inch in diameter and 1.25 inches long.
[2] 3 percent H_2O.
[3] Some spotty or irregular attack.

Experiments on Oxidation of Nickel-Chromium Alloys in Air

In the first series of tests in air, the nickel-chromium alloys listed in Table 2 were exposed for four days at 1900 F. The test specimens were 0.375 by 1 by 0.03 inch in size and were laid four at a time in a quartz tube inserted in a tubular electric furnace. Air was passed through the quartz tube.

The weight gains in Table 2 showed the expected behavior. According to the literature, the addition of chromium to nickel does not improve the resistance to oxidation until 8 to 10 percent is present.[9,10] Thick black oxide scales formed on alloys containing up to about 9 percent chromium. With more chromium, the character of the scale changed; it became thin, grey colored and protective with low weight gains. The change occurred at about 9 percent chromium with this composition showing erratic behavior.

Micro-examination disclosed that internal oxidation had taken place in the alloys containing up to 9 percent chromium. Figure 3 illustrates the behavior of alloys containing 3, 6 and 9 percent chromium. In the figure, the thick external scales had spalled away. The internal oxidation of the 9 percent chromium alloy occurred only under the areas coated with thick oxide. There was no internal oxidation of this alloy under areas coated with the thin protective oxide. The thick external oxides had two zones: a coarsely-crystalline, columnar outer zone and a fine, apparently granular inner zone. The dividing line was at the position of the original surface. The thick scales undoubtedly were essentially nickel oxide with some chromic oxide dissolved in the lattice. The oxide at the scale-metal interface supplied enough oxygen to support the internal oxidation. Micro-examination of the alloys high in chromium showed negligible sub-surface attack.

The second series of tests in air were conducted on a larger number of alloys. To get all these in the constant temperature zone of the furnace at the same time, the holder shown in Figure 4 was constructed. The specimens were rods 1.25 inches long and 0.1 inch in diameter. The spacers were held against the central shaft of the holder by set screws. Holes were drilled through the spacers parallel to the shaft to allow free passage of gases through the holder.

The alloys in this test are listed in Table 3. One series contained 4 to 20 percent chromium. Another series contained 14 percent chromium with a wide range of iron contents. Several alloys corresponded to Inconel except that substantial amounts of copper, molybdenum, silicon, columbium or titanium had been added.

The results after eight days of exposure at 1750 F are included in Table 3. At this temperature the oxidation was less than at 1900 F but showed the same characteristics. Alloys with 4 to 8 percent chromium had comparatively high weight gains, and micro-examination showed internal oxidation. This is illustrated in Figure 5 where the external oxide is still in position. Alloys high in chromium showed negligible attack.

Experiments in Dry Carbon Monoxide

The Research Laboratory of the Mond Nickel Company at Birmingham, England, found that internal oxidation could be produced in some nickel-chromium alloys by exposure to carbon monoxide.[11] The alloys in Table 3 were exposed to carbon monoxide, taken directly from commercial cylinders of this gas for 1, 4, 8 and 16 days at 1750 F. The holder shown in Figure 4 was used, and the carbon monoxide was passed through the quartz tube of the furnace at the rate of 150 cc per minute and burned as it left the furnace. This was essentially the procedure used by the Mond Nickel Company.[11]

Severe carburization and internal oxidation occurred in some of the alloys. Generally, the attack increased with time although there were some irregularities. The results at eight days are representative and are included in Table 3. The weight gains in the table give a rough indication of the relative severity of the attack. When severe attack occurred, there was some increase in diameter of the rods although this was only a few mils at the most.

Figures 6, 7, and 8 show the progression of the attack in the alloy containing

14 percent chromium, balance nickel. Severe internal oxidation developed, and there was carburization ahead of this. The alloy with 20 percent chromium, balance nickel, behaved similarly. With less chromium (4 and 8 percent) the internal oxidation was not as deep, the oxides were not as massive and no carburization was detected ahead of the internal oxidation. However, the internal oxidation in these alloys was deeper in the tests in carbon monoxide than in air.

The addition of 6 percent iron to the alloy containing 14 percent chromium, balance nickel, decreased the internal oxidation. The effect of more iron was rather uncertain, the results being erratic. The alloy containing 20 percent chromium and 20 percent iron was resistant to attack. The addition of copper or of molybdenum to the 6 percent iron, 14 percent chromium alloy did not have much effect. Figure 9 shows a cross section of the alloy containing copper, etched to bring out the carburization ahead of the oxidation. Magnification is higher than in Figures 6, 7 and 8.

The presence of 2 percent silicon helped to decrease both carburization and oxidation. The addition of the carbide stabilizers, columbium and titanium practically stopped all attack. This is illustrated in Figure 10 for the alloy containing 2 percent columbium. No carburization was detected in this alloy. The effect of columbium in stopping this kind of attack has been patented.[12]

Experiment in Wet Carbon Monoxide

An experiment was made in which the carbon monoxide was saturated with water at room temperature before it entered the furnace. The carbon monoxide contained an estimated 3 percent water vapor. Otherwise the test conditions were the same as before. The results after eight days at 1750 F are included in Table 3.

One effect of the moisture was to increase the depth of internal oxidation in the low chromium alloys. Possibly the moisture catalyzed or increased the carburization although as before no carburization was detected in those low chromium alloys. Figure 11 shows the attack in the case of the alloy containing 8 percent chromium. The preference of the attack for the grain boundaries is to be noted.

The alloy containing 20 percent chromium, balance nickel, showed the most severe internal oxidation produced in any of these tests. The attack is shown in Figure 12. There was considerable increase in diameter with the result that cracks developed as shown in the figure.

The behavior of the other materials in this test was the same as in dry carbon monoxide.

Discussion

Laboratory tests showed that in oxidizing environments some internal oxidation of low-chromium nickel alloys is to be expected. With high-chromium nickel alloys, however, a thin protective external scale forms, and there seems to be no internal oxidation unless some complicating factors come into play.

Rapid internal oxidation of high-chromium alloys was produced by simultaneous exposure to carburizing and oxidizing conditions. The formation of carbides produces chromium depletion in the surrounding matrix, but at the high temperatures involved, recovery by chromium diffusion would be fairly rapid so that severe attack requires practically simultaneous carburization and oxidation. Evidently the carburization need not be severe because in some cases none was detected, the important consideration being chromium depletion.

In this respect, internal oxidation is analogous to intergranular corrosion of stainless steels in aqueous solutions. The analogy is made stronger by the beneficial effect observed for additions of columbium and titanium although here, of course, the action is confined to the surface.

Oxidizing conditions normally form protective oxide coatings on high chromium alloys so that it may be difficult for internal oxidation to start. Once internal oxidation is well under way, however, the volume increase and swelling would prevent the formation of a protective external scale in marginal atmospheres. The laboratory tests have indicated that the addition of such elements as columbium and titanium, which form carbides more stable than chromium carbide, is an effective means of preventing this type of internal oxidation.

Pure carbon monoxide at high temperature is not often encountered commercially, but it is often a constituent in controlled atmospheres in furnaces. Obviously more work is required to define the ranges of gas mixtures which can be considered safe or dangerous. The effect of temperature on the rate of attack should be determined.

Examination of specimens removed from service indicates that carburization is not the only cause of internal oxidation in high-chromium nickel alloys. More research is required to find all the conditions which may cause this attack. Fortunately, internal oxidation of nickel-chromium alloys has been comparatively rare under actual operating conditions.

Acknowledgment

The authors wish to acknowledge indebtedness to R. F. Stern, who ran some of the preliminary experiments, and to G. R. Pease, R. A. Kozlik and others at the Research Laboratory who examined service specimens.

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Any discussion of this article not published above will appear in the December, 1959 issue

NATIONAL ASSOCIATION of CORROSION ENGINEERS

Identification of Corrosion Products On Copper and Copper Alloys

A Report of NACE Technical Unit Committee T-3B on Corrosion Products*

NACE TECHNICAL COMMITTEE REPORT
Publication 59–13

Abstract

A description is given of micro-chemical analytical methods that can be used to identify corrosion products on copper and copper alloys. Both soluble and insoluble constituents can be identified by these methods. Constituents discussed in detail include oxides, chlorides, sulfates, sulfides, carbonates, silica, calcium, magnesium, sodium, ammonia, and metal constituents of the material being examined.

A method for spectrographic examination of corrosion products is considered briefly.

3.4.3

Introduction

THE MAJORITY of copper alloys submitted for examination of corrosion products will have been exposed to some type of outdoor atmosphere, fresh water, or sea water. Normally, the corrosion products are thin, tightly adherent layers. Usually, the following constituents are determined either qualitatively or quantitatively:

Oxides	Magnesium
Chlorides	Sodium
Sulfates	Ammonia and also
Sulfides	the metal constituents
Carbonates	of the material being
Silica	examined.
Calcium	

Silica, carbonates, sulfates, chlorides, and salts of calcium, magnesium, and iron are generally found on any object exposed to any type of natural atmosphere or water.

In the atmosphere, a layer of cuprous oxide usually will be found next to the metal with mostly basic copper sulfate in the top layer of corrosion products. Occasionally, basic copper carbonate and cupric oxide, in small amounts, will also be found. In industrial atmospheres, sulfides may be present. In marine atmospheres, basic copper chloride may be mixed with the basic sulfate.

In fresh water, cuprous oxide is found next to the metal surface, particularly in pits. Basic copper carbonate is often a constituent of this corrosion product. Cupric oxide may be found on metals exposed to hot water.

In sea water, cuprous oxide is usually present next to the metal surface, with basic copper chloride in the top layer of the corrosion product. In addition, cuprous sulfide and some basic copper carbonate may be found on copper alloys exposed to polluted sea water.

X-ray and electron diffraction methods are sometimes used in conjunction with chemical methods to determine constituents in corrosion products. The chemical methods used can be found in standard references for qualitative and quantitative analysis. The names of several publications covering methods of analysis that are adapted to determining corrosion products are listed in the attached bibliography.

Micro-chemical analytical methods such as those described below are most useful for determining the nature of corrosion products, especially when only a small sample is available. The metallic components of corrosion products can be quickly determined by emission spectrographic analysis, but this approach does not give information on the compounds present. For quantitative spectrographic data, synthetic standards must be prepared. The accuracy of the spectrographic analysis depends on the experience of the spectrographer.

Method for Spectrographic Examination

A 20 mg sample is weighed into a chemically pure graphite cup electrode and burned to completion in a 10 ampere direct current arc with the sample serving as the anode. A rotating sector at the spectrograph slit is adjusted to pass about 8 percent of the light. The prism or grating is adjusted to record wave lengths within the region of 2500 to 3500 A, which includes the persistent lines for most of the common elements. Secondary lines of calcium, sodium, and potassium also will be recorded.

Methods for Micro-chemical Examination

Character of Corrosion Products

In the preliminary examination of the corroded sample, the physical appearance, such as thickness, texture and colors, of the corrosion product should be noted. All these are helpful in identifying constituents and applying subsequent micro-chemical tests. Immersing the specimen in a solution of concentrated ammonium hydroxide containing about 10 percent (by weight) ammonium chloride or in hydroxylamine hydrochloride will remove most of the corrosion products at a rate slow enough that periodic examinations can be made, thereby revealing conditions which otherwise would escape notice. Hot hydroxylamine hydrochloride removes corrosion products rapidly.

Water Washing to Collect Soluble Constituents

The area of the specimen needed for examination is computed to a fraction of a square foot. Visible oil film is removed with petroleum ether or other suitable solvent and the surface washed down with boiling distilled water. The amount of wash water to be used will depend on the size of the sample and the nature of the corrosion product. A 1 inch square sample having a relatively large amount of corrosion products may require only about 50 ml of fresh water, while a sample with an area of 30 square inches and having a relatively thin, dense film of corrosion products might require 2 liters of wash water. The washings are filtered through a 15 cm diameter No. 40 Whatman filter paper and evaporated to a volume of 1 to 2 ml. Where large volumes of wash water are involved, washings are evaporated down to about 100 ml in a large beaker and then transferred to a 150 ml beaker for the final concentration. The concentrated wash-

* A. H. Roebuck, The Western Company, Midland, Texas.

ings are transferred to a 15-ml centrifuge tube and centrifuged. The volume of the solution is recorded in tenths of a milliliter.

Determination of Soluble Constituents

Determination of soluble constituents is made by adding the proper chemical to one drop of the concentrated water washings. In the majority of cases, reagents are added in a solid form. Silver nitrate and barium chloride, which are most frequently used, are more conveniently added in liquid form. A 10 percent (by weight) solution is used, added as a micro-drop (about 0.01 ml.) In many cases the precipitate can be identified without the use of a microscope; in others, its use is required for confirming the structure of the precipitate.*

Total Solids. A drop of the solution (0.05 ml) is transferred to a 1 by 3 inch glass slide and evaporated to dryness. The residue is compared with a series of standard residues mounted between glass containing about equal calcium acetate, sodium chloride, ammonium chlorides, hydrated calcium sulfate and silica. Each standard residue is double the amount of the previous one, and is in the range from 0.00002 gm to 0.0064 gm. The amount of solids in the unknown is matched with one of the standard residue and the total solids are calculated in grams per square foot.

pH. This is determined on one drop using indicator paper or a liquid indicator.

Chlorides. Solution of silver nitrate is added to one drop of concentrated water washings.

Sulfates. Solution of barium chloride is added to one drop of concentrated water washings.

Sodium. One drop is evaporated to dryness. Solid uranyl acetate is added and the residue covered with 1:1 acetic acid. After standing for 10 or 15 minutes the substance is examined for crystals of sodium uranyl acetate.

Calcium and Organic Matter. One drop of washings is evaporated to dryness. The residue is covered with 1:4 sulfuric acid and heated to fuming. In the presence of fixed organic matter discoloration may occur or the drop may become very black. 1:4 nitric acid is added and the solution heated to destroy color. The fuming is continued until all free sulfuric acid is removed. The drop is taken up in water and after 1 to 3 minutes is examined for hydrated calcium sulfate.

Magnesium. The drop from above, after examining for hydrated calcium sulfate, is diluted, made ammoniacal and calcium precipitated as the oxalate. It is filtered off and the filtrate tested for magnesium with di-sodium phosphate. If zinc is absent, magnesium may be detected in the presence of calcium sulfate by making ammoniacal and adding di-sodium phosphate.

Copper and Zinc. A drop is evaporated, organic matter destroyed using sulfuric acid and nitric acid and all sulfuric acid removed by fuming. The residue is then flooded with 1:4 sulfuric acid and mercuric potassium thiocyanate added. Copper forms green crystals while those of zinc are white or lavender tinted.

Silica. In making tests for calcium, involving fuming with sulfuric acid, silica is dehydrated with much of it clinging to the glass. The dehydrated silica is very conspicuous. To confirm the presence of silica, a drop of solution is placed on a ⅞ inch diameter plastic cover, ammonium fluoride and sodium chloride added and the solution made acidic with the addition of sulfuric acid. Silica forms hexagonal crystals of sodium fluosilicate having a low refractive index and exhibiting a faint pink color.

Ammonia. Solutions of ammonium chloride are prepared equivalent to 0.00000165 gm, 0.0000033 gm, 0.0000066 and 0.000013 gm ammonia per drop of 0.05 ml.

A drop of the washings is treated with one small drop of 10 percent platinum tetrachloride and heated gently until a crust begins to form around edge. The solution is examined at 75X and compared with drops of the standard solutions. Potassium salts must be absent as well as organic nitrogen bases.

If potassium is present, a large drop of the solution should be placed in a small porcelain crucible, a drop or two of 1/10-normal sodium hydroxide solution added to the crucible, and the liquid covered with a glass slide having a drop of dilute hydrochloric acid suspended on its lower surface. The crucible should be warmed gently and allowed to stand 15 to 30 minutes. Ammonia will collect in the hydrochloric acid solution and can be tested for by the procedure outlined above.

If it is suspected that amines are present, a drop of the solution should be evaporated to dryness with concentrated hydrochloric acid and leached with absolute ethyl alcohol. The alcohol will extract the chloride of the amine, but not the ammonium chloride. A test should be made for ammonia in the residue.

Other Cations. In making the original evaporation of washings, the presence of iron and silica is usually evident; the former imparts a brown color or actually separates as a flocculent precipitate; the silica sticks to wall of beaker and also separates as gel. Potassium may be detected with perchloric acid. Appropriate tests are made for any other cation suspected of being present or for anions other than chloride or sulfate.

Examination of Insoluble Constituents

Silica. Silica is usually present and may be soft and loose and easily brushed away or it may be tightly adherent. Both kinds are frequently found on one surface. Silica can be recognized by its appearance but may be confirmed by converting to sodium fluo-silicate using ammonium fluoride, sodium chloride and sulfuric acid.

Iron Oxide. Iron is usually recognized by its color which varies from light brown to almost black. Placing a drop of potassium ferrocyanide acidified with hydrochloric acid on the surface usually produces the color of Prussian blue.

Magnetic Iron Oxide (Fe_3O_4). The presence of this oxide is indicated by its magnetic property.

Cuprous Oxide. The physical appearance of cuprous oxide is unmistakeable and is sufficient identification. Confirmation can be obtained by moistening with acetone acidified with hydrochloric acid which converts the oxide to white cuprous chloride.

Cuprous oxide occurs as garnet-red crystals, as a glassy garnet-red coating or as a powdery rust-colored hydrated oxide.

Cupric Oxide. The black film should be flooded with cold 1:1 sulfuric acid. The cupric oxide will dissolve and the color change to that of copper. Another possibility is to place a drop of warm hydroxylamine hydrochloride solution on the black film. Cupric oxide will be dissolved by this solution.

Zinc Salts. A piece of the metal sample is immersed in a saturated solution of hydrogen sulfide in 10 percent acetic acid. Zinc sulfide will appear as a white cloud falling away from the surface of the metal. This occurs before copper salts start to fall away. Alternatively, scrapings may be tested for zinc using mercuric potassium thiocyanate and sulfuric acid, which will form white or lavender colored crystals.

Carbonates. The presence of carbonates is indicated by the evolution of gas when a drop of dilute acid is placed on the surface of the specimen. This can be confirmed by holding a drop of dilute sodium carbonate solution containing phenolphthalein in the gas evolved. The solution will be decolorized if the gas is carbon dioxide.

Tin or Tin Oxides. The surface of the specimen is flooded with a solution of sodium cyanide and allowed to stand for five minutes. A check should then be made for a metallic tin coating at the treated area. This method is particularly useful for finding tin compounds in pits in copper alloys of which tin was a constituent. Treating a specimen with 40 percent acetic acid solution containing an equal volume of 6 percent hydrogen peroxide solution will dissolve copper salts but leave metallic tin or tin oxides intact.

* A precipitate may form during evaporation, but this should not cause concern because any constituent in the precipitate will still be present in the concentrated solution. The formation of a precipitate does, however, give an indication that certain constituents, such as sulfates, carbonates and silica, are present.

Aluminum Compounds. Several small pieces of the sample are placed in 10 percent hot sodium hydroxide solution. The solution is then centrifuged and placed on a glass slide. The solution is acidified with sulfuric acid, and a lump of cesium chloride added. The glass slide is rubbed with a glass rod to aid the formation of octahedral cesium aluminum chloride compound which is colorless.

Nickel. Nickel alloys may be oxidized; if so the oxide may in part be green-tinted nickelous oxide or a mixture of this green and the black oxide (nickelic oxide). Both can be recognized by their appearance but the presence of nickel should be confirmed with dimethylglyoxime.

Mercury. This contaminant is deleterious to copper alloys and causes intergranular cracking. It can be detected in many instances by visual inspection after dipping the sample in 1:1 nitric acid. Much smaller amounts of mercury can be detected by putting a convenient sized sample into a 10 to 15 cc porcelain crucible covered with a glass slide. A beaker of cold water is placed on the glass to act as a condenser. The crucible is placed in a hole in a shielding asbestos sheet, and the bottom of the crucible heated with a pilot flame for about 10 minutes. Mercury will be transferred to the glass where it can be seen. The presence of mercury can be confirmed by converting the material collected on the slide to red mercuric iodide. Cyano iodide or a soluble iodide can be used as the reagent.

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Evaluation of Steam Resistant Coatings For Carbon Steel Hospital Ware*

By E. G. BOBALEK, W. R. R. PARK, E. G. BELL and W. R. DAWSON

Abstract

Steam exposure presents the most severe environment for producing blistering failure of synthetic resin coatings. Conflicting evidence has been presented over the past several years regarding the performance of modern coatings in preventing the corrosion of steel in steam exposure. Tests were made to determine the feasibility of replacing stainless steel hospital ware with coated carbon steel ware. In this connection an experimental survey was made by panel testing more than 120 commercial coatings representing about 25 different chemical types over a variety of surface treatments. Tabular comparisons were made regarding the relative worth of different types of coatings. It was found that coated mild steel could not be regarded as an adequate substitute for stainless alloys in the manufacture of sterilizers. It was discovered, though, that a number of coating types, properly applied, could withstand 500 hours or more exposure to 250 F steam.

5.4.8

Introduction

MANY ARTICLES of hospital ware now being supplied to the armed services are fabricated from stainless alloys. The weight of these items ranges from fractions of an ounce to hundreds of pounds each. For example, one portable field sterilizer unit requires about 400 pounds of stainless steel. This raw material is expensive and fabrication costs are high, particularly in complicated shapes. These disadvantages of stainless steel are tolerated because the durability of the products is outstanding.

Although stainless steel gives the most satisfactory service performance, the Military Medical Supply Agency[1] initiated a program to evaluate substitute materials. Decreased availability of stainless alloys during periods of national emergency could necessitate some decisions regarding use of alternative materials which are in more abundant supply. Mild steel would provide the most convenient substitute for stainless alloys if its corrosion resistance could be sufficiently upgraded. Such coatings would have to withstand high pressure steam because hospital materials are sterilized by autoclaving. Durability of a paint film during steam exposure presents an unusual and severe specification. Information regarding this property of coatings is not delineated clearly in the literature.

This part of the program was not concerned with development of new steam-resistant coatings, but rather with a comprehensive evaluation of commercial coatings. This evaluation was made to determine whether the coating industry could now provide materials for upgrading the corrosion resistance of mild steel sufficiently to permit its use in hospital equipment. For this evaluation a field sterilizer was singled out as a specific development target. Any coating which could provide adequate protection for a carbon steel sterilizer might be expected to give good protection to many other items which ordinarily are exposed to less severe service conditions. Most items of hospital ware are not required to withstand autoclaving for the very long periods required of the sterilizer itself.

Experimental Work

Sample Procurement

To assure a representative sampling of potentially steam-resistant paints, two procedures were followed. First, five producers of coatings were contacted and invited to participate through their own laboratories in the evaluation procedure. These five suppliers provided samples of such formations as they judged would have the best chance of success as steam-resistant coatings; in some cases, experimental and as yet non-commercial formulations were submitted for examination.

The second group of samples was obtained from a large number of manufacturers whose advertisements claimed special corrosion-resistant properties for their products. In this broad sampling, even such products as appeared to be only slightly suitable were ordered and evaluated. This extensive sampling was done to minimize the chance that useful materials might be excluded because of prior prejudice regarding their chances of being steam-resistant.

Using information provided by the suppliers and/or the authors' analytical data, the paint samples were classified into groups according to the dominant resin type of the formulation. This classification of paints into broad chemical categories did not make fine distinctions between specific formulations; actual formulation details were never obtained since the manufacturers were naturally reluctant to part with classified information. Hence in each category, the paints might be expected to show a wide range of quality because of formulation variables.

Application Procedures

All of the screening tests for potentially steam-resistant protective coatings were carried out on mild steel panels which had a zinc phosphate surface treatment (Parker Spra-bond 100).

Wherever possible, the paint application was made by dipping at a uniform speed, using a motor-driven pulley adjustable to give dipping rates ranging from 1"-10" per minute.[2] This application method had the advantage of coating both sides of the panel simultaneously and also gave a more uniform and reproducible film thickness per dip than could be obtained by spraying. In a multiple coat operation the wedge effect of a single dip coat can be compensated by reversing the panel for each successive dip.

At least six panels were coated with each coating formulation. Three of the six panels were coated (multiple dips) to a thickness of about 2 mils while the other three received 4-6 mils of coating. Four of these, two of the heavy and two of the light film thicknesses were then given the final bake recommended by the manufacturer of the coating. The remaining two panels were baked at a temperature higher than that recommended by the supplier as standard for the coating.

Exposure

Before the coated panels were exposed to steam, half of the prepared set from each formulation were given a 180 degree bend around a half inch diameter mandrel and also given two cuts, down to base metal, about 2 inches long and which intersected at right angles. These operations gave an indication of the flexibility, hardness, brittleness and adhesion of each coating prior to steam exposure. The panels of each set, except for control blanks, were hung in a surgical sterilizer and exposed to 20 psi steam. The panels were observed frequently for signs of degradation by comparing with the control panels. Panels were removed from test exposure either when the coating was judged to be no longer serviceable, or after 500 hours of exposure, whichever came first.

Those coatings which maintained protective value after 500 hours of steam exposure test were compared in detail

★ Submitted for publication March 3, 1958. A paper presented at a meeting of the North East Region, National Association of Corrosion Engineers. Pittsburgh, Pennsylvania, November 12-14, 1957.

[1] This study was performed under the industrial mobilization program of the Military Medical Supply Agency of the Department of the Navy which has the single manager assignment for medical materials for the Army, Navy and Air Force. The work described herein is part of the survey of the problem of replacing stainless alloys with coated mild steel in a variety of hospital ware if military necessity should ever require the use of alternate materials to conserve the supply of certain materials.

[2] W. R. R. Park and W. R. Dawson. Constant Speed Dipping Devise. *Paint & Varnish Production Magazine*, (1957) June.

Bobalek

Park

Bell

E. G. BOBALEK is Professor of Chemical Engineering, Case Institute of Technology, where he is in charge of the coatings and plastics research laboratories. He specializes in research on theoretical problems of engineering application of polymeric materials.

W. R. R. PARK, a graduate of University of Toronto, came to Case as a Research Associate after two years as Research Chemist with Canadian Industries Limited. In the period of July 1955 to February 1957, Dr. Park was in direct charge of the experimental work reported in this paper. Dr. Park is presently with the Dow Chemical Company at Midland, Michigan.

E. G. BELL joined the Case Research Staff after three years as Research Chemist with Canadian Industries Limited, where he specialized in the formulation and testing of paints and synthetic resins. He is presently a graduate student at Case Institute where his research work is involved in the application of radioactive isotopes in latex emulsion problems.

W. R. DAWSON, a graduate of Case Institute of Technology, is an off campus candidate for a doctorate degree in physical chemistry. He is presently with the Research Laboratories of Republic Steel in Cleveland, Ohio.

TABLE 1—Non Steam-Resistant Coatings

Chemical Type	Film Thickness (Mils)	Final Bake	Hours to Failure	Comments
Alkyd	4-6	20 Minutes at 400F	7.5	Film cracked and blistered badly; in some cases became very tacky.
Silicone	2.0	30 Minutes at 450F	23	Softened badly.
Vinyls	4	15 Minutes at 450F	65	Softened badly and completely lost adhesion.
Rubber base Coatings: Chlorinated rubber——→	5.5	10 Minutes at 350F	30	Blistered badly.
Synthetic rubber——→	16	900 Minutes at 200F	8	Softened and swelled badly—low adhesion.
Bituminous	9	240 Minutes at 250F	280	Softening and interlaminar blistering.
Urethane	4	60 Minutes at 285F	19	Serious loss of adhesion.
Teflon	4	.....	65	Bad underrusting and blistering; film remained unchanged.

with unexposed panels of the same formulation. The properties measured were: hardness, adhesion, abrasion resistance, stain resistance, color retention, gloss retention, edge rusting, underrusting at scratch and impact resistance (Appendix 1.) Panels which failed at less than 500 hours' exposure to steam were sometimes given a second chance if it appeared that application variables might be the cause of early failure. Otherwise, the mode of failure was noted and the panels were discarded from further consideration.

Discussion of Panel Testing Results

The majority of the coatings which failed to pass the 500 hour test failed after less than 50 hours' exposure. Many of the chemical classes which included some formulations that passed the 500 hour exposure test also contained some specific formulations which failed in less than 50 hours. It cannot be assumed that a coating will be steam-resistant simply because it happens to be one of a class which contains steam-resistant coatings. Each prospective paint, whatever its composition, must be tested separately. In general, however, some chemical classes contained no steam-resistant formulations at all, and if any formulation is known to be of that class, it might be discarded without further testing with a very small chance of error.

Discussion

Many types of protective coatings now in use employ resins which contain ester linkages in their structures. This accounts largely for the relatively low hydrolytic resistance of these materials. This class of saponifiable coatings and also such non-saponifiable thermoplastic coatings which have softening points below the 250 F steam sterilization temperature were eliminated *a priori* from the evaluation. The reasons are discussed here why certain resin systems cannot be expected to give satisfactory performance in high pressure steam.

Non-Steam Resistant Types

Alkyds. Although this type of material is widely used in the appliance and automotive industry (modified with either melamine formaldehyde or urea formaldehyde resins) it does not have good steam resistance; the ester groups in the molecules are susceptible to hydrolysis. This leads to rapid softening and general degradation of this type of paint on exposure to steam.

Silicones. Straight or modified silicones have been very successful in applications that require a highly heat-resistant paint; however, the silicon-oxygen bond present in all silicone resins has a low degree of resistance to hydrolysis at elevated temperatures. As a result, this type of coating, while excellent in hot dry applications, fails rapidly in a hot and wet environment.

Vinyls. Most vinyl-type resins fail rapidly because of thermoplastic softening of the coatings at the test temperatures. Steam diffuses readily through the soft film and disintegrates the adhesion bond to the substrate.

Rubber Base Coatings. Coatings based on natural, synthetic, chlorinated and sulfur (types of) rubber all absorb water and soften badly during exposure to steam. As a result of the high absorption the coatings soften and swell and the adhesion bond is destroyed prematurely. It is doubtful that this type of coating ever could be upgraded sufficiently to have good steam-resistant properties.

Miscellaneous Types. Urethane coatings show premature failure, probably due to hydrolysis of both ester and urethane linkages.

Bituminous coatings soften rapidly in the test conditions and rapidly lose their protective properties.

Teflon coatings have the ultimate in chemical resistance of all the synthetics available to the authors. However, both pigmented and clear Teflon films applied from dispersion formulations are microscopically porous and allow for rapid under-film corrosion.

Flame sprayed nylon coatings undergo physical or chemical changes during steam exposure which cause them to embrittle badly and lose adhesion.

Table 1 contains a summary of test results of the non-steam resistant types.

Steam Resistant Types

Modified Phenolics. The utmost caution must be exercised in the formulation of steam resistant phenolics; the films suffer severe dimensional stresses on exposure to steam. The over-all result of these stresses is to weaken or to break the adhesive bond leaving the substrate open to attack.

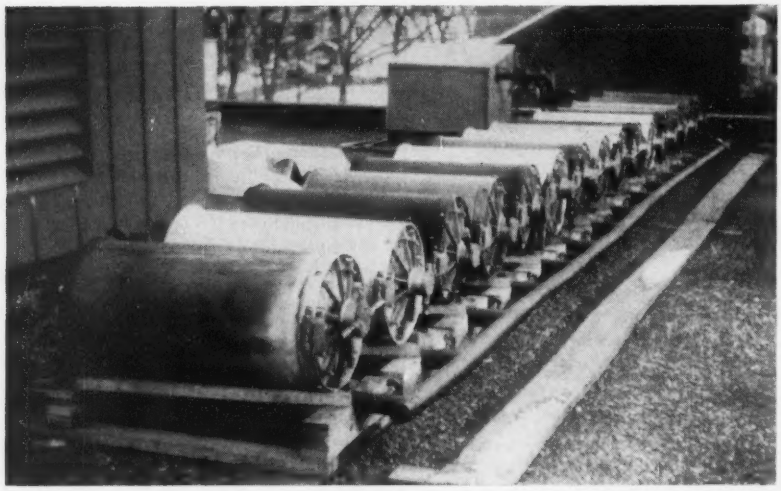

Figure 1—Battery of sterilizers under test at Case Institute of Technology. The timer is enclosed in the box in the center background and the steam line runs parallel to the rear of the units. There is a 1 psi differential in the steam pressure between the first and last sterilizer.

Figure 2—Exposed sterilizer with a section cut out of the outside wall to allow examination of the annulus.

The majority of coatings in this class were commercial products. These are, undoubtedly, modified with small quantities of other resins but no detailed information could be obtained on these additives. Thus, it was not known how the dimensional stability of the steam resistant phenolics was obtained. However, several formulations based on Bakelite Resin BR 17620 blended with polyvinyl butyral displayed good properties.

Modified Expoxies. While this class of coating produced several very steam resistant types (epoxy-phenolic), other modifications such as epoxy esters, nitrogen resin-epoxy blends, epoxy-polyamide, epoxy thiokol showed only poor to fair steam resistance. The main weakness of these types of formulations appears to be the sensitivity of the modifying agents; when the additives suffer degradation the entire performance characteristics of the coating are altered and degraded.

Miscellaneous Types. Several miscellaneous coatings showed good steam resistance. Butarez 15, a heat-curing polybutadiene formulation had good ratings in all tests, probably because of a lack of polar groups (focal points for steam attack) in the cured resin. The main objections to this coating are the difficulty in application and the high temperature (500 F) required for adequate curing.

Formvar, a polyvinyl formal-phenolic blend used for wire enameling also showed good properties. Unfortunately, the high viscosity of the solution and the toxicity of the phenolic solvents which it employs, limit the usefulness of this material. It is too difficult and hazardous to handle to be of practical value for the application here studied.

Kel-F coatings (polymonochlorotrifluoroethylene) gave the best performance of any organic coating tested. As might be expected, it is also by far the most expensive material examined from the standpoint of both material cost and application cost.

In all, 120 protective coatings representing 30 chemical types were evaluated; only 6 of these chemical types (represented by 11 coatings) showed sufficient resistance to degradation by steam to warrant further evaluation.

Table 2 contains a summary of the properties of the steam-resistant coatings; a rating scale of 0-10 has been used: 10—excellent, 8—very good, 6—good, 4—fair, 2—poor, 0—very poor.

Effect of Coating Variables on Steam Resistance

Final Film Thicknesses

In general, the heavier film thicknesses were more prone to blistering than the lighter film thickness.

Final Bake Conditions

Formulations containing polyvinyl butyral showed lower steam resistance properties when the films were baked at temperatures above 350 F. On the other hand, epoxy phenolic films seemed to reach an optimum cure at 425-475 F. This range is 25-100 degrees higher than usually recommended by the manufacturer. The coatings thus obtained generally are less flexible than if cured at the recommended schedule but the increased cure leads to better over-all properties for this application.

Variations of Surface Treatments

An evaluation of the available methods for substrate preparation was carried out with a view toward obtaining the best

TABLE 2—Steam-Resistant Coatings

Chemical Type	PROPERTY													
	Steam Resistance	Hardness	Stain Resistance	Chemical Resistance	Impact Resistance	Flexibility	Abrasion Resistance	Undercut Resistance	Blistering	Adhesion	Ease of Application	Color Retention	Total*	Estimate of Relative Cost†
Phenolic	8	7	9	9	1	1	9	8	9	4	10	5	80	1.7
Epoxy Phenolic	8	6	8	7	6	7	7	8	7	8	10	8	90	2.0
Phenolic PVB	6	5	7	4	6	6	7	7	5	7	10	3	73	1.8
Polybutadiene	8	6	8	8	6	8	8	6	9	7	9	8	91	1.8
Kel-F	9	5	10	10	10	10	9	8	9	7	2	9	98	11.0
Epoxy Polyamides	8	7	8	8	5	5	4	9	9	7	8	8	86	2.1

* Perfect score = 120
† Cost in dollars/ft² of applied finish at 4-6 mils thickness divided by cost of 4-6 mils of standard alkyd type of appliance enamel (refrigerator finish).

TABLE 3—Field Test of Coated Sterilizers

REF.	Coating Type	Hours Exposure at 20 PSI Steam					
		1000	2000	3000	4000	5000	6000
4	Aluminum dipped mild steel	No change	Slight rusting on back of body	Increased rusting on back of body	**Failed**—the seam split		
5	Nickel plated mild steel	Slight rusting on back plate	Slight rusting on body	Same	Same	Same	Same
6	Porcelain	No change	Severe erosion over entire body	Very severe erosion	Same	Same	Same
7	Aluminum	Slight oxide coating	Heavy white oxide coating	Same (only exposed 3000 hrs. to date)			
8	No treatment	Severe rusting	Formation of magnetic iron oxide	Same	Same	Same	Same
10	Kel-F	Rusting at back plate and front edge	Bad discoloration over entire body	Loss of adhesion around front edge	Same	Same	Same
11	Epoxy-phenolic	Slight blistering at front edge	Slight flaking at front edge	Loss of adhesion on back plate	Same	Same	near **Failure**
12	Modified phenolic	**Failed**—blistered and peeled					
13	Epoxy phenolic	Bad chalking	Same	Flaking and rusting along front edge	Same	Same	Bad rust collection
14	Epoxy phenolic	Bad blistering at back plate and front edge	Checking on back plate	Same	**Failed**—flaking over body		
15	Epoxy phenolic	Bad blistering and rusting at front edge	Cracking around back plate	Same	Same	Bad flaking at back plate and front edge	
16	Phenolic	Blistering and flaking on back plate	Flaking on body	**Failed**—checked and flaked over entire body			
17	Modified epoxy	**Failed**—loss of adhesion between topcoat and primer					
18	Polybutadiene	Checked over back plate	**Failed**—checked and flaked over entire body				
19	Modified epoxy	Slight blistering	**Failed**—blistered over entire body				
20	Modified epoxy	Slight blistering at front edge	Bad discoloration over entire body	Blistering over back plate	Bad under-rusting at front edge and back	Same	Same
21	Modified phenolic	No change (only exposed 1500 hrs. to date)					

4. A. Tickle Engineering Works, Brooklyn, New York.
5. Kanigen Division, General American Transport Corporation. The Kanigen coating is chemically deposited using a 92-8 alloy of nickel and phosphorous.
6. Heintz Manufacturing Company, Philadelphia, Pennsylvania.
7. Aluminum Company of America 6061 T fabricated by American Sterilizer Company, Erie, Pennsylvania.
8. A. C. Lab Equipment Company, Bronx, New York.
10. Kel-F, manufactured by the M.W. Kellogg Company, Jersey City, New Jersey, and applied by the Falls Hollow Staybolt Company, Cuyahoga Falls, Ohio.
11. Kordell 600 green, manufactured by the Kordell Industries, Chicago, Illinois, and applied by Kordell Industries, Mishawaka, Indiana.
12. 20% XYSG and 80% BR17620 (Bakelite Company, a division of Union Carbide Corporation). The formula was developed at Case Institute of Technology, Cleveland, Ohio, and applied by the Arco Company, Cleveland, Ohio.
13. Landau White, Grade II, manufactured by the W. J. Landau Company, New York, and applied by Metalweld, Inc., Philadelphia, Pennsylvania.
14. General Resistant Coating, manufactured by the Monsanto Chemical Company and applied by Geddes Custom Coaters, Cleveland, Ohio.
15. SP4376, manufactured by the Specialty Coatings Incorporated, (division of Thompson Company), Oakmond, Pennsylvania, and applied by the Arco Company, Cleveland, Ohio.
16. P413 and L66, manufactured and applied by the Heresite & Chemical Company, Manitowoc, Wisconsin.
17. Dennis No. 1150 and No. 1100, manufactured and applied by the Dennis Chemical Company, St. Louis, Missouri.
18. Butarez 15, manufactured by the Phillips Petroleum Company, Bartlesville, Oklahoma, and applied by the Geddes Custom Coaters, Cleveland, Ohio.
19. Trailite No. 2, manufactured and applied by R. J. Grant Company, Compton, California.
20. Arco LS40489, manufactured and applied by the Arco Company, Cleveland, Ohio.
21. Plastite No. 7155, manufactured by the Wisconsin Protective Coating Company, Green Bay, Wisconsin. The coating arrangements were made by the manufacturer.

possible performance from a given coating material. Conventional types of treatments (iron phosphated steel, sandblasted steel, vapor degreased steel, and galvanized iron) were tried in addition to treatments that modified the surface character either by deposition of another substance or by controlled magnetic iron oxide formation on the surface. These included treatments with graphite base coatings, tetra isopropyl titanate, nickel ammonium sulfate, and oxidation in molten nitrite and nitrate, dichromate salt baths. No advantage was seen, however, from the use of these alternate methods as compared to the inexpensive and readily applied zinc phosphate coatings.

Flame sprayed coatings of aluminum oxide and zirconium oxide were examined as adhesion promoters and as protective film.[3] Excellent metal protection was observed when the oxide surface was impregnated who phenolic type coatings; however, the coating was judged unsatisfactory in this application because of poor flexibility and high application costs.

Field Service Program

The obvious extension of the panel testing program was to test, under actual service conditions, coatings which performed favorably. Additional systems not examined in the original evaluation were included here; these were (a) an aluminum-dipped mild steel sterilizer,[4] (b) a nickel-plated mild steel sterilizer (using only 1/20 the nickel requirements of a stainless steel unit and no chromium),[5] (c) a porcelain-coated mild steel sterilizer,[6] (d) an all aluminum sterilizer.[7]

The first problem that had to be solved was that of obtaining a satisfactory phosphate coating on the specially fabricated mild steel sterilizer shells.[8] As a rule, custom paint applicators do not have phosphating tanks large enough to handle such big pieces of equipment; this meant that the phosphating and coating operations had to be carried out at different locations. Therefore, methods of preserving the condition of freshly applied phos-

[3] Norton Manufacturing Company, Worcester, Massachusetts.
[4] A. Tickle Engineering Works, Brooklyn, New York.
[5] Kanigen Division, General American Transport Corporation. The Kanigen coating is chemically deposited using a 92-8 alloy of nickel and phosphorous.
[6] Heintz Manufacturing Company, Philadelphia, Pennsylvania.
[7] Aluminum Company of America 6061 T fabricated by American Sterilizer Company, Erie, Pennsylvania.
[8] A. C. Lab Equipment Company, Bronx, New York.

phate coatings, while in transit, had to be examined. It was found the surface condition was preserved by sealing the freshly phosphated sterilizer with a non-corrosive dessicant and some paper impregnated with a vapor phase inhibitor into a disposable polyethylene bag.

To ascertain that all coatings received the same surface preparation, the mild steel sterilizer shells were taken to specialists for the phosphating treatment. After each unit was treated, it was dried and cooled and packaged in a polyethylene bag; each bagged unit was packed carefully in soft paper packing before crating to prevent punctures of the polyethylene bags.

The units were then expressed to the respective manufacturers of such coatings which had shown a high degree of steam resistance in the panel screening tests. Arrangements were made by the paint suppliers to have the sterilizers painted according to their recommended specifications; the coatings were applied to both the inside and outside surfaces of the sterilizer shells under the supervision of the technical personnel of the coating manufacturer. The insides of the annular steam jackets were acid cleaned and coated with a graphitic-base steam boiler tube lining.[9]

Sixteen units, consisting of 11 shells coated with the organic coatings and one shell protected by each of the following treatments were mounted on the roof of the chemistry building at the Case Institute of Technology: (a) procelain coated, (b) nickel plated, (c) aluminum dip, (d) all aluminum, (e) one untreated control unit. Figures 1 and 2 show views of the sterilizers. The units operated under 20 psi steam using a two hour on, one hour off cycle for 17 hours a day, 5 days a week until cold weather made it necessary to change the cycle (trouble was encountered with steam lines freezing). The cycle was then changed to 1¾ hours on, ¼ hour off 24 hours a day, 7 days a week. This cycle was continued until warmer weather made it possible to revert to the original cycle.

The exposure was started in the fall of 1956 and to date the units have been exposed to intermittent live steam for approximately 6000 hours. The condition of the coatings was examined after every 200 hour exposure interval. When the degree of failure was considered severe enough to make the unit unserviceable, the sterilizer was removed from service. Thus far, seven of the units have been removed; coatings on remaining units show various degrees of degradation.

Table 3 contains the history of the coatings up to 6000 hours' exposure. Performance records are listed at 1000 hour exposure intervals.

[9] Axpexior No. 1, Dampsey Manufacturing Company, Boston, Massachusetts.

Conclusions

For various reasons summarized in Table 3 coated mild steel cannot be regarded as an adequate substitute for stainless alloys in the manufacture of sterilizers. However, as compared to the situation of a few years past, it is surprising what enormous advances have been made in the design of formulations which are steam resistant. Ten years ago, the capacity of a coating to withstand 20-50 hours of steam exposure at 250 F would have been regarded as an outstanding performance. Some of the more modern coatings give good, even if not perfect, protection for several thousand hours under conditions of intermittent steaming and cooling. (This is a much more severe test than continuous exposure to steam.)

Many of the failures of the coated, carbon steel sterilizers can be explained by failure to accomplish ideal conditions of paint application, in general. Coated carbon steel stands a better chance of competing with stainless alloys if the coated units are small and simple in design. For example, items such as wash basins, instrument trays, bed-pans and miscellaneous hospital ware are hardly any more complicated to finish properly than are test panels, and the total service life exposure to high temperature steam will be only a fraction of what is expected of the sterilizer itself. For such more modest requirements, a number of coatings of types listed in Table 3 should provide adequate competition to the stainless alloys now used in most conditions of service.

The effectiveness of zinc phosphating as a metal surface treatment for steam resistant paints is somewhat contrary to much popular opinion. The only possible equal is sandblasting followed by immediate coating. However this is, in general, more difficult to control under production conditions. The preparations of metal for adhesion promotion is one of the most important directions that should be investigated in subsequent research on steam resistant coatings.

This comprehensive evaluation of commercial coatings undoubtedly is not complete. However, it is adequate to demonstrate which general trends of resin formulation are likely to show the most promising results. Further upgrading of the coatings of these chemical types should be possible by: (a) improving curing uniformity at variable film thicknesses, and (b) exclusion of all traces of water sensitive ingredients from the formulation. Presently, however, the variables caused by imperfect control of paint application, under shop conditions, is important enough to hide relatively minor variations of steam resistant qualities of the better coatings.

APPENDIX 1—Test Proceedings

(a) Scratch Test: The unexposed coated panels were given two crossed two-inch cuts to the metal with a sharp knife. Any chipping about the scratch was noted.

(b) Bend Test: Each coated panel was given a 180 degree bend around a half-inch mandrel; any cracking, peeling or loss of adhesion was noted.

(c) Impact Test: This test consisted in determining the maximum impact in inch-pounds from a half-inch diameter steel ball that a coated panel would withstand without cracking or loss of adhesion in the coating film.

(d) Gloss Measurement: The gloss measurement rating is effectively ten times the percent of light that is reflected from the paint surface at an angle of reflection of 60 degrees where the angle of incidence is also 60 degrees. The measurements were made on a Gardner 60 degree Glossmeter.

(e) Abrasion Test: This test was performed by rotating No. 15 or No. 17 abrasive wheels against a rotating paint surface on a Taber Abrasion Tester. The abrader head carried 2000 grams for all tests. The results were reported as loss of coating weight in milligrams per 1000 revolutions of the paint panel; in addition each coating was rated after 100 and 1000 revolutions.

(f) Microknife Adhesion Test: The instrument, the Arco Microknife, uses a conical shaped diamond cutting head which carries weights from 0-1000 grams. A measure of the adhesion of the coating was obtained by measuring the distance between parallel cuts to the metal, in mils, that caused flaking of the coating between the cuts.

(g) Microknife Hardness Test: The Arco Microknife can be used also to obtain relative values for the hardness of coatings; this was done by noting the number of strokes that were required for the weighted diamond cutting tool to cut through to the substrate and also noting the weight carried by the diamond head.

(h) Staining Test: Ammoniated mercury and chlorinated hydroxyquinoline ointments, as well as a few drops of a saturated solution of iodine in ethyl alcohol, were applied to three separate spots on the exposed and unexposed panels. A cap was placed over each of the materials to prevent evaporation; after 24 hours the panels were wiped clean, washed with acetone, and inspected for signs of staining.

(i) Film Thickness Determination: The film thickness measurements were made with a General Electric Electromagnetic Film Thickness Meter; several readings were taken over the panels and the values obtained were averaged.

Any discussions of this article not published above will appear in the December, 1959 issue

Delayed Failure of High Strength Steels*

By ALEXANDER R. TROIANO

Abstract

High-strength steels plated with cadmium for protection against corrosion were found to fail unexpectedly at high stress levels. A definite connection between this phenomenon and the introduction of hydrogen into the steel through pickling and plating was determined. Tests were made to determine the effect of hydrogen concentration and notch radius on the delayed failure of aircraft quality SAE-AISI 4340 steel samples. As might be expected, hydrogen concentration exerted a potent influence on the static fatigue curve.

The kinetics of crack initiation and propagation were studied by an electrical resistivity method. The existence of an incubation period for crack initiation and a period of apparently controlled crack growth were apparent. Data on the reversibility of the incubation period were reported.

Baking to eliminate hydrogen from steel was effective with thin plates, but thickly plated parts still exhibit embrittlement. A new recovery treatment was developed to solve this problem without increasing baking times on temperatures for thicker plates. This method involves double electroplating of cadmium and depends for its effectiveness on the ability of cadmium to delay the passage of hydrogen. 3.2.2

Introduction

MANY STEEL parts require corrosion protection even from normal atmospheres. This is particularly true of the highly stressed high-strength steel components of modern aircraft. Paint would serve this purpose, but the problem of virtually constant maintenance, protection against scratching and peeling and inaccessability make paint quite unsatisfactory.

Cadmium plating provides excellent permanent protection without maintenance or susceptibility to mechanical damage and has been used with success on steels at low to medium strength levels. However, at the high strength levels demanded by modern design (225,000 to 275,000 psi), cadmium plating appears to introduce the potentiality for brittle delayed failure in service.

This is a particularly insidious type of failure in that spontaneous brittle fracture may occur at loads substantially below those that may have been previously sustained and in material which shows good ductility in conventional tests. For example, electroplated steel bolts, heat treated to high strength levels, have shown delayed failure at the root of threads or in the head fillet. Cadmium plated aircraft components, such as landing gear struts, have failed in a completely brittle manner while under very low stresses; yet when the material of the fractured component is subsequently tested, it indicates full normal high ductility.[1,2,3,4] This same phenomenon is present under certain conditions in titanium alloys as well as steel.[5]

Recently a definite association has been realized to exist between this phenomenon and pickling and plating which introduces hydrogen into the steel.[6]

Using these observations, Case Institute of Technology investigated the influence of cadmium plating and hydrogen on the mechanical behavior of aircraft quality 4340 steel, heat treated from 200,000 to 280,000 psi tensile strength.[(1)] Some of the results of this study are presented.[7]

Results and Discussion

Throughout this study two types of tensile specimens were used. Their general shape and significant dimensions are indicated in Figure 1.

Both notched and unnotched tensile specimens were cadmium plated and treated to obtain a uniform concentration of hydrogen. This was possible because the cadmium plate strongly retards the outgassing of hydrogen. The sequence was (1) electrolytic charge in sulphuric acid, (2) cadmium plate and (3) bake at 300 F.

The hydrogen content could be changed by varying the baking time. This is demonstrated by the data of Figure 2. This figure indicates that the hydrogen concentration is uniform because there was no change in ductility with successive removal of material from the unnotched specimens in the baked material as compared to the as-plated specimens.[(2)] The changing level of ductility, however, clearly indicates that the hydrogen concentration is reduced as the baking time is prolonged. See Figure 3.

The method of studying delayed failure was simply to load statically a series of sharply notched tensile specimens at different stresses and wait for failure. Delayed failure was observed over a wide range of applied stresses. Figure 4 depicts the type of curve obtained for a specific hydrogen content corresponding to that obtained by baking 3 hours at 300 F.

About the Author

ALEXANDER R. TROIANO—Head, Dept. Metallurgical Engineering, Case Institute of Technology, Cleveland. Dr. Troiano, active in research connected with phase transformations in metals, on which he has published some 50 papers, has a PhD from Harvard Graduate School of Engineering Metallurgy. He has been an instructor at Middlesex College, Mass.; at Harvard University and Notre Dame, Indiana and has held his present post at Case since 1953. His recent work has been in high strength steels and titanium alloys. He has received numerous awards and honors for his work in metallurgy, including the AIME Hunt Medal, ASM Howe medal and is active in six professional engineering organizations. He is consultant to many industrial organizations and to the U. S. armed forces.

There are a number of significant features that should be noted in this figure:

1. The notch tensile strength is less than normal and directly reflects the loss of ductility due to hydrogen embrittlement.

2. Delayed failure occurred over a wide range of applied stress.

3. The rupture time is only slightly dependent upon the applied stress.

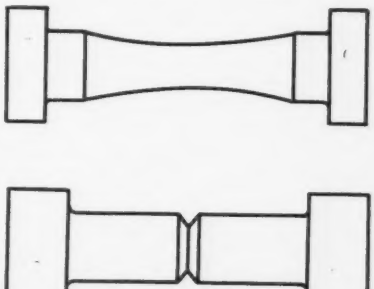

Figure 1—Tensile Specimens. Top specimen is unnotched (1½ inches long, 2-inch radius, 0.212-inch diameter reduced section). Bottom specimen is notched (1½-inches long, 0.212-inch notch diameter, 50 percent-60 degree sharp notch).

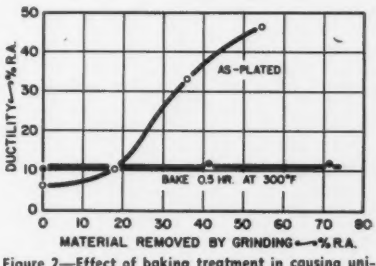

Figure 2—Effect of baking treatment in causing uniform hydrogen distribution.

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(1) This study was made possible by the support of the U. S. Air Force, through the Wright Air Development Center, Dayton, Ohio.

(2) Since hydrogen analyses do not show significant differences, it is necessary to assume that hydrogen content and ductility are directly related. This is a generally accepted premise for the ductility ranges considered here.

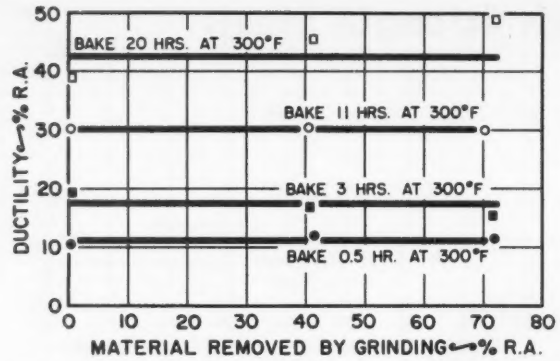

Figure 3—Variation of hydrogen concentration level with baking time at 300 F.

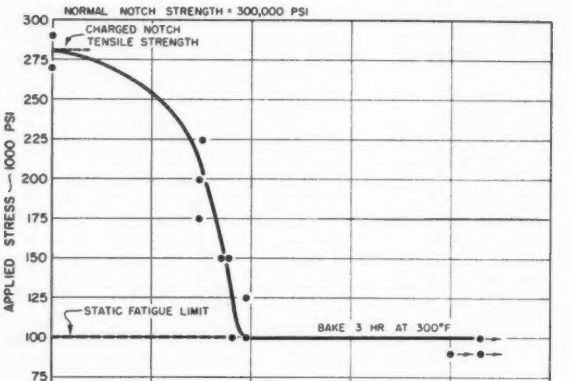
NORMAL NOTCH STRENGTH = 300,000 PSI
CHARGED NOTCH TENSILE STRENGTH
STATIC FATIGUE LIMIT
BAKE 3 HR AT 300°F
APPLIED STRESS — 1000 PSI
FRACTURE TIME — HOURS

Figure 4—Static fatigue curve for sharp notch specimens hydrogenated, cadmium plated and baked 3 hours at 300 F at 230,000 psi strength level.

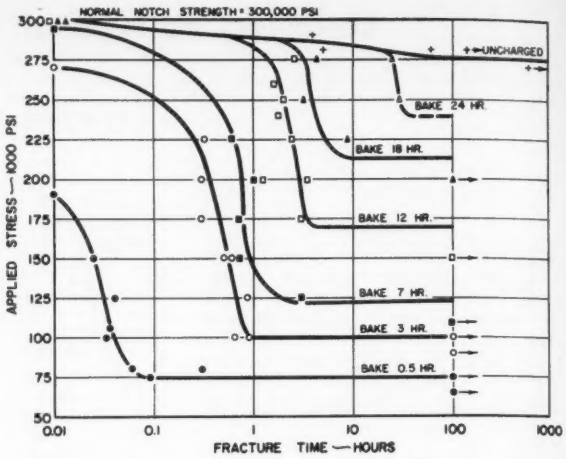

Figure 5—Static fatigue curves for various hydrogen concentrations corresponding to different baking times at 300 F. Sharp notch specimens were used at 230,000 psi strength level.

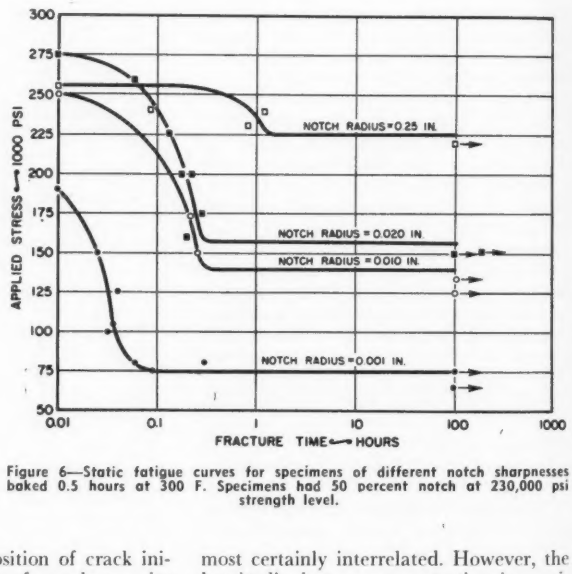
NOTCH RADIUS = 0.25 IN.
NOTCH RADIUS = 0.020 IN.
NOTCH RADIUS = 0.010 IN.
NOTCH RADIUS = 0.001 IN.
APPLIED STRESS — 1000 PSI
FRACTURE TIME — HOURS

Figure 6—Static fatigue curves for specimens of different notch sharpnesses baked 0.5 hours at 300 F. Specimens had 50 percent notch at 230,000 psi strength level.

4. There is a lower critical stress below which failure will not occur, presumably for any length of time. (A static fatigue limit.)

As might be expected, hydrogen concentration exerts considerable influence on the static fatigue curve.

Static fatigue curves for hydrogen concentrations (baking times) are indicated in Figure 5. All delay failure parameters, *notch strength, rupture time* and *static fatigue limit,* increase continuously with decreasing hydrogen concentration.

The increase in lower critical stress with decreasing hydrogen concentration suggests that delayed failure (crack initiation) is controlled by a combination of hydrogen and stress. With decreasing hydrogen concentration, more stress is necessary to initiate delayed failure. This concept is supported by experiments on specimens of varying notch severity.

The variation of the lower critical stress with notch severity is shown in Figure 6. Static fatigue limit rises as the notch severity (radius) decreases. It is significant also that the stress concentration correlates so well with the static fatigue limit (Figure 7).

Additional support is obtained from an examination of the position of crack initiation as a function of notch severity (Figure 8). Sharp notch specimens are observed to crack slightly below the notch bottom.[7] These results agree qualitatively with the concept that the most severe triaxial stress state exists below the notch surface and that the position of maximum triaxiality moves into the specimen as the notch severity is decreased.[8, 9]

At this point it is advisable to note that stress concentration and triaxiality are not physically equivalent quantities. Stress concentration refers to the increase in *longitudinal* stress resulting from structural or geometrical imperfections. Triaxiality is a three dimensional stress state in which all stresses are tensile. The difference between the two quantities can be emphasized by noting that a triaxial stress state cannot be created in a two dimensional specimen where a stress concentration can.

In a three dimensional specimen, triaxiality and stress concentration are almost certainly interrelated. However, the longitudinal stress concentration is maximum at the specimen surface whereas experimental evidence (photoelastic stress analysis) indicates that triaxiality occurs below the notch surface.

The correlation between point of crack initiation and location of maximum triaxiality suggests that triaxiality, rather than stress concentration, is the dominating factor.

Initiation, Propagation and Fracture

Attention is given here to the events occurring prior to fracture in the static fatigue range. The cracks shown in Figure 8 and other evidence indicate that delayed failure occurs by a process of crack initiation and relatively slow propagation.

The kinetics of crack initiation and propagation were studied by an electrical resistivity method developed at Case.[10] A typical resistivity-time curve for sharply notched specimens is shown in Figure 9. The existence of an incubation

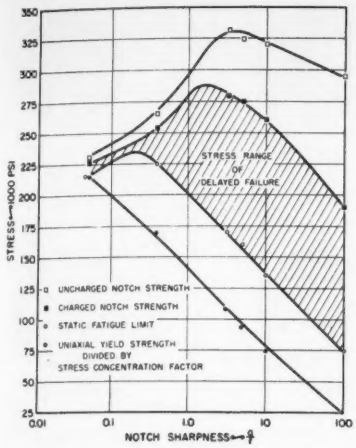

Figure 7—Embrittlement parameters as a function of notch sharpness. In the ratio $\frac{a}{r}$, "a" is half diameter of notch cross section; "r" is notch radius.

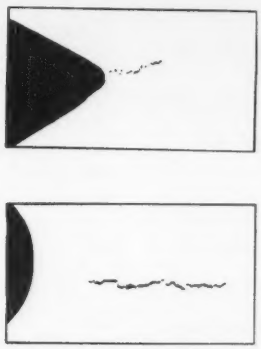

Figure 8—Cracks observed in notch specimens sectioned after static loading. Specimens were hydrogenated, cadmium plated and baked at 300 F. Longitudinal section at 100X. Top specimen has 0.001-inch notch radius, 12-hour baking time and 240,000 psi applied stress. Bottom specimen has 0.010-inch notch radius, 0.5-hour baking time and 225,000 psi applied stress.

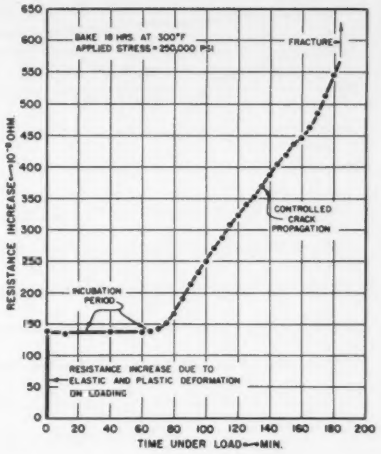
BAKE 18 HRS. AT 300°F
APPLIED STRESS = 250,000 PSI
FRACTURE
RESISTANCE INCREASE—10⁻⁹ OHM
CONTROLLED CRACK PROPAGATION
INCUBATION PERIOD
RESISTANCE INCREASE DUE TO ELASTIC AND PLASTIC DEFORMATION ON LOADING
TIME UNDER LOAD—MIN.

Figure 9—Typical resistivity-time curve for sharp-notched specimens at 230,000 psi strength level.

TABLE 1—Reversibility of the Incubation Period*

Loading Time (min) at 250,000 psi	Aging Time	Incubation Period (min) on Reloading at 250,000 psi
17	1 min	8
20	24 hr	18
20	24 hr	30
20	24 hr	30

* Normal Incubation Period =30 min.

period for crack initiation and a period of apparently controlled crack growth are apparent. The existence of an incubation period (seconds to days) is a matter of considerable importance. For example, one of the unique characteristics of hydrogen embrittlement is that the fracture ductility decreases with decreasing strain rate.[11] This fracture ductility is dependent upon both initiation and propagation of a crack; nevertheless, for a given incubation period, a low strain-rate test will more likely detect embrittlement than a high strain-rate test. If the strain-rate is so high that the specimen fractures in less time than the incubation period, hydrogen embrittlement will not be detected.

This behavior must be reflected in the design of a suitable test for detection of hydrogen embrittlement. Apparently, a mechanical test will be inadequate if the test duration is less than the incubation period. Since incubation periods of substantial duration have been observed, short time tests such as normal tensile and bend tests may not be discriminating. Thus, under these conditions, the steel may exhibit normal ductility but will still be subject to brittle delayed fracture.[12]

Figure 10 shows that the incubation period is strongly dependent upon hydrogen but that applied stress has little influence. The incubation period is interpreted tentatively in the following manner.[7,13] Just prior to testing, the hydrogen is uniformly distributed throughout the specimen. The proper or critical combination for failure of the hydrogen concentration and stress does not exist in the region of high triaxiality since the specimen does not fail on loading; but since the load or stress does not change, the incubation time must be that time necessary for the hydrogen to concentrate in the region of maximum triaxiality. The concentration of hydrogen is considered to be induced by the high elastic strain in the region of high triaxiality. When sufficient hydrogen has concentrated in this region, a crack will be initiated.

Since it has been postulated that the diffusion of hydrogen is elastic strain-induced, it follows that the incubation period should be stress reversible. In other words, the direction of hydrogen diffusion should reverse upon removal of the stress. Stress-induced diffusion should result in a concentration peak at the point of most severe triaxial stress. When the applied stress is removed, the uniform distribution should be restored after a suitable aging time.

This reversibility feature is vividly demonstrated in Table 1. Sharp notch specimens were treated to a hydrogen concentration corresponding to an incubation period of about 30 minutes. The specimens were then stressed at 250,000 psi for the indicated times which were shorter than the incubation period, unloaded and aged at room temperature.

Specimens aged for 24 hours exhibited normal incubation periods upon reloading; the specimen unloaded for one minute displayed a much shorter incubation period because the aging time was insufficient to allow hydrogen diffusion.

Because the incubation period is reversible, total incubation periods much longer than the normal incubation period may be produced by alternate loading and aging treatments (Figure 11). A total incubation period of 82 minutes is produced for a normal incubation period of 30 minutes and a total incubation period of 200 minutes is produced for one of 55 minutes. The 10-minute incubation period indicates that the aging treatments do not cause any detectable out-gassing which might influence the results.

The electrical resistivity curve of Figure 9 and also heat tinting experiments indicate that the period of slow crack growth is followed by catastrophic failure with extremely fast crack growth. The appearance of the fracture differentiates between the area of slow crack growth and that of catastrophic failure (see Figure 12).

1. The average slow crack propagation rate can easily be measured as the slow propagation crack area divided by the difference between rupture time and incubation period.

2. This average crack propagation rate is independent of applied stress and strongly dependent upon hydrogen concentration (Figure 13).

3. The fracture stress was constant and approximately equal to the true (unembrittled) fracture stress of the material. This was true over a wide range of hydrogen concentration and applied stress. This means that the crack grows slowly at a rate controlled by hydrogen diffusion to the crack tip until the true fracture stress of the material is exceeded.

Thus the variation in rupture time reflects the total time for slow crack growth to attain the critical area where L/A equals fracture stress. Fracture stress is believed to be constant and equal to the unembrittled (non-hydrogenated) fracture stress because catastrophic failure involves very high strain rates. At very high strain rates, hydrogen embrittlement is not normally evident, however, this may not be the situation at extremely high hydrogen concentrations.

Recovery of Cadmium Plated Embrittled Steel

A relation apparently exists between delayed failure and the reduced ductility (embrittlement) caused by the introduction of hydrogen by pickling and cadmium plating.

To mitigate hydrogen embrittlement, a baking treatment is often specified which is designed to recover ductility by driving the hydrogen from the steel.[2,3,4] For a

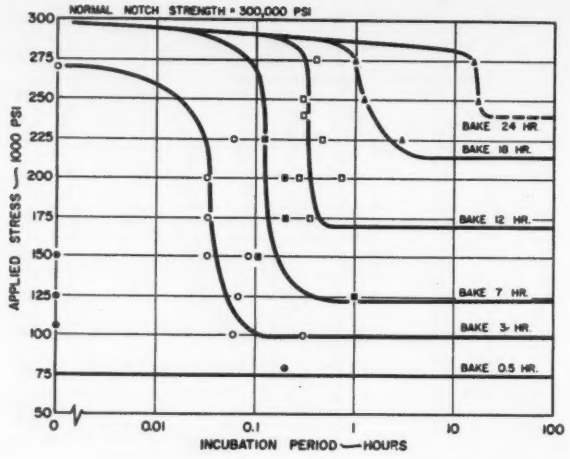

Figure 10—Variation of incubation period wtih applied stress and hydrogen concentration corresponding to different baking times at 300 F. Specimens were sharp-notched, 230,000 psi strength level.

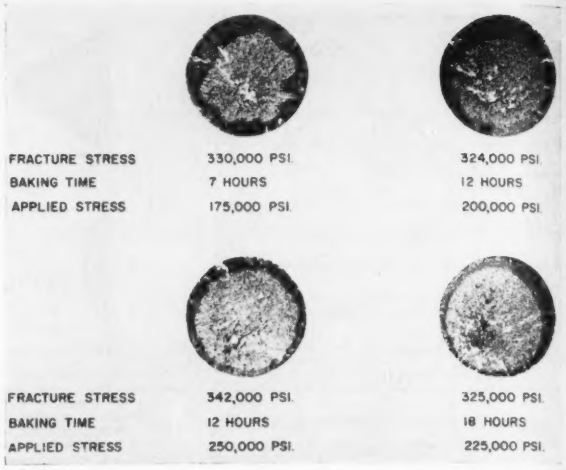
FRACTURE STRESS 330,000 PSI. 324,000 PSI.
BAKING TIME 7 HOURS 12 HOURS
APPLIED STRESS 175,000 PSI. 200,000 PSI.
FRACTURE STRESS 342,000 PSI. 325,000 PSI.
BAKING TIME 12 HOURS 18 HOURS
APPLIED STRESS 250,000 PSI. 225,000 PSI.

Figure 12—Fracture surface of sharp notched specimens hydrogenated, cadmium plated and baked at 300 F. Dark areas indicate extent of hydrogen controlled crack propagation. Strength level was 230,000 psi, 7.5X.

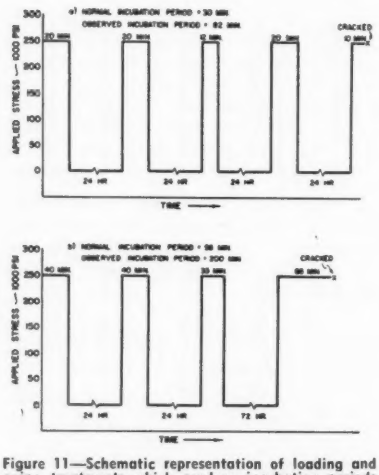

Figure 11—Schematic representation of loading and aging treatments which produce incubation periods much longer than the normal incubation periods for these hydrogen concentrations. Sharp notch specimens at 230,000 psi strength level.

TABLE 2—Composition of Cadmium Plating Solution

CdO	NaCN	NaOH	Brightening Agent
32 gm/1	112 gm/1	12 gm/1	4.5 gm/1

TABLE 3—Barrier Effect of Cadmium Plate

	Bake 0.5 Hrs at 300F	Bake 3.0 Hrs at 300F
Sequence 1(De-Plated)	40% R.A.	40% R.A.
Sequence 2(Plated)	10.5% R.A.	17% R.A.

Sequence #1	Sequence #2
a)Hydrogen Charge	a)Hydrogen Charge
b)Cadmium Plate	b)Cadmium Plate
c)De-Plate (Chromic Acid Solution)	c)
d)Bake at 300F	d)Bake at 300F

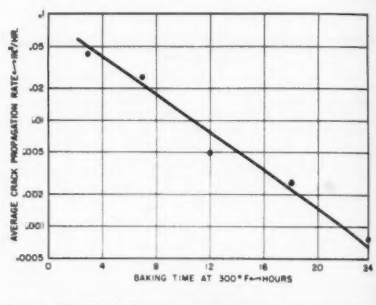

Figure 13—Variation in average crack propagation rate with baking time at 300 F. Sharp-notched specimens at 230,000 psi strength level.

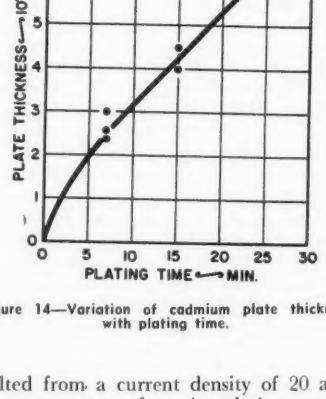

Figure 14—Variation of cadmium plate thickness with plating time.

cadmium plated part, a typical recovery treatment consists of baking for several hours in the 300 F to 400 F temperature range. However, the incidence of brittle failures in electroplated parts demonstrates that baking treatments are not always effective. Several factors are considered to influence the efficiency of a recovery treatment; only a few of these have been investigated. For a given baking treatment, there is less recovery of ductility as the strength level of the base steel increases. Also, the recovery process follows a time-temperature sequence because greater recovery is achieved from higher baking temperatures or longer baking times. Other factors of significance have not been investigated. For example, it has not been demonstrated whether hydrogen outgassing is governed primarily by the base steel or the cadmium plate. If the cadmium plate is the controlling factor, then a question of vital interest is the influence, if any, of plate thickness upon the recovery rate. An examination of these factors has led to a new and promising technique for minimizing hydrogen embrittlement.[14]

The material employed was the same aircraft quality SAE-AISI 4340 steel examined in the delayed failure experiments. The study was conducted with the steel heat-treated to 230,000 and 270,000 psi tensile strength. The 2-inch radius tensile specimen shown in Figure 1 was employed.

The sequence of events in the plating operation was designed to simulate commercial treatment of high strength steels, as follows: (1) electrolytic hydrogenation, (2) cadmium electroplate and (3) bake at 300 F or 400 F.

Initially, the specimens were degreased in carbon tetrachloride and then cathodically charged for five minutes in a 4 percent sulfuric acid solution at a current density of 1.4 x 10^{-4} amperes per square foot. This step simulates, in a reproducible manner, the acid pickling or other surface cleaning treatments given commercial parts before electroplating.

The composition of the cadmium plating bath is given in Table 2.

A bright, adherent cadmium plate resulted from a current density of 20 amperes per square foot. A cadmium anode was used and the plating bath was not agitated.

The cadmium plate thickness was conveniently controlled by varying the plating time, given in Figure 14. It should be noted that the plate thicknesses cover the range specified by the ASTM.

That the cadmium plate controls hydrogen outgassing is demonstrated by a comparison of the recovery characteristics of plated and of plated plus deplated specimens, given in Table 3. Two groups

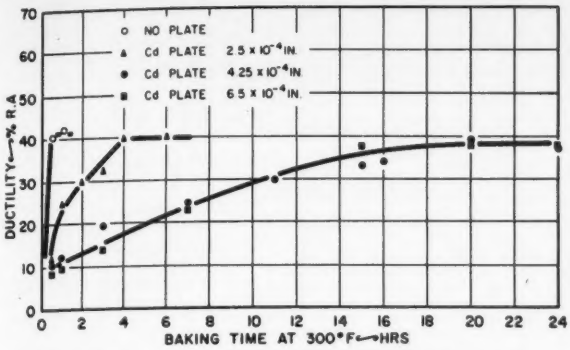

Figure 15—Recovery behavior for various cadmium plate thicknesses.

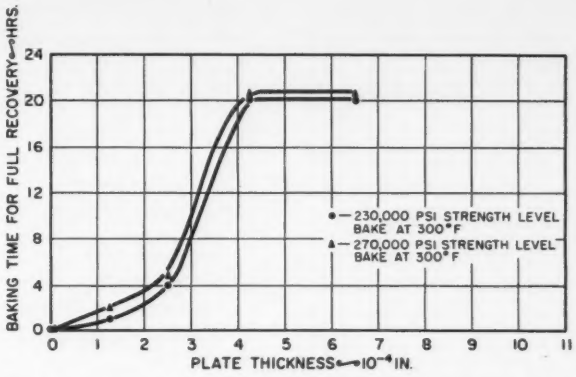
BAKING TIME FOR FULL RECOVERY→HRS.
●—230,000 PSI STRENGTH LEVEL BAKE AT 300°F
▲—270,000 PSI STRENGTH LEVEL BAKE AT 300°F
PLATE THICKNESS→10⁻⁴ IN.

Figure 16—Influence of plate thickness on baking time necessary for complete recovery of ductility.

of specimens at the 230,000 psi strength level were electrolytically charged and cadmium plated under identical conditions; the cadmium plate was removed from one group of specimens by dipping in a chromic acid solution just prior to the baking treatment.

The cadmium plate is a potent deterrent to recovery since the deplated specimens exhibit complete recovery while the plated specimens are severely embrittled. Evidently, the cadmium delays the passage of hydrogen and therefore is an efficient barrier to hydrogen outgassing.

If this behavior is considered, it is not surprising to find that the cadmium plate thickness strongly influences recovery as documented in Figure 15. The same behavior pattern is evident upon baking at both 300 F and 400 F although recovery is slightly faster at 400 F. In both cases, thinly plated specimens recover rapidly; thickly plated specimens may require as long as 20 hours or more baking for complete recovery.

These data allow an evaluation of typical commercial baking treatments for recovery. Commercial baking treatments are evidently effective with thin plates; thickly plated parts may still exhibit severe embrittlement. Because of this, inordinately long baking times may be necessary to obtain complete recovery as indicated in Figure 16. Within the range of commercial plate thicknesses, the necessary baking time varies from a few hours to as long as 20 or more hours. Of course, these baking times depend upon the particular embrittlement conditions, and these specific values are valid only for the hydrogen charging conditions employed. In service, additional factors such as section size, strength level, and stress raisers may influence the recovery behavior; even longer baking times than reported here might be necessary for complete recovery. The effect of these factors would presumably be additive to that of plate thickness.

This influence of plate thickness upon recovery is undoubtedly responsible for much of the current confusion regarding hydrogen embrittlement in service. Because of the different operating conditions likely to be found among electroplating shops and the normal variations in plate thickness over irregularly shaped parts, wide variations in embrittlement may be expected.

This phenomenon poses a dilemma: requirements for maximum corrosion resistance and for minimum hydrogen embrittlement are mutually incompatible. A reasonably thick plate is necessary for good corrosion resistance; a thin plate is desirable to minimize hydrogen embrittlement.

The problem then is to deposit a thick electroplate and, at the same time, obtain full recovery after a reasonably short baking treatment. An obvious approach would be to use higher baking temperatures; however, this is limited by two factors: the susceptibility of cadmium to oxidation and the original tempering temperature of the base steel to obtain the high strength levels.

A new recovery treatment has been developed at Case which depends upon the ability of cadmium to delay the passage of hydrogen. The basis of this treatment is cited in Figure 17 which represents the variation of ductility with plating time. The ductility decreases rapidly to a limiting value during the first few minutes of plating and then remains constant.[3] Evidently, hydrogen enters the steel during the first few minutes of plating and cannot enter after a critical thickness of plate metal has been deposited.

An apparently efficient recovery treatment was devised by taking advantage of this behavior. The procedure is as follows: a thin layer of cadmium is electroplated on the steel which is then baked to recovery. The baking time is short because the plate is thin. A second layer of cadmium—of any thickness—is then deposited. After this second plating, no embrittlement is observed because the first plate prevents the entry of hydrogen. This technique is so efficient that no baking treatment was necessary after the second electroplating. The validity of the method for both strength levels and several plate thicknesses is demonstrated in Figures 18 and 19.

As an example, a cadmium plate of

[3] This limiting ductility does not result from saturation with hydrogen because much lower ductilities have been observed.

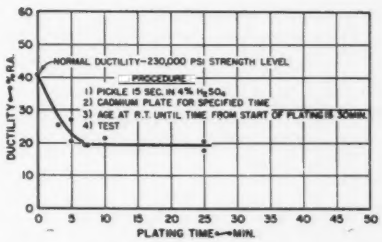
NORMAL DUCTILITY—230,000 PSI STRENGTH LEVEL
PROCEDURE
1) PICKLE 15 SEC. IN 4% H₂SO₄
2) CADMIUM PLATE FOR SPECIFIED TIME
3) AGE AT R.T. UNTIL TIME FROM START OF PLATING IS 30MIN.
4) TEST
DUCTILITY ←% R.A.
PLATING TIME←MIN.

Figure 17—Influence of plating time on ductility.

about 7.5 x 10^{-4} inch is to be deposited on steel heat treated to 230,000 psi. In this case Figure 16 would be applicable. First a cadmium plate of 1.25 x 10^{-4} inch is deposited. The steel is then baked to full recovery, requiring only one hour at 300 F. A second cadmium plate of 6.5 x 10^{-4} inch—or any other thickness—may then be deposited and no embrittlement will be observed. On the other hand, if the standard sequence of charging, plating and a three-hour bake at 300 F had been employed, the ductility would have been reduced to 17 percent area reduction, which is less than one-half the normal ductility of 40 percent area reduction.

This method has considerable potential as a commercial recovery treatment and should be of particular value in critical applications where maximum corrosion resistance is desired, with a minimum of hydrogen embrittlement and concomitant susceptibility to brittle delayed failure.

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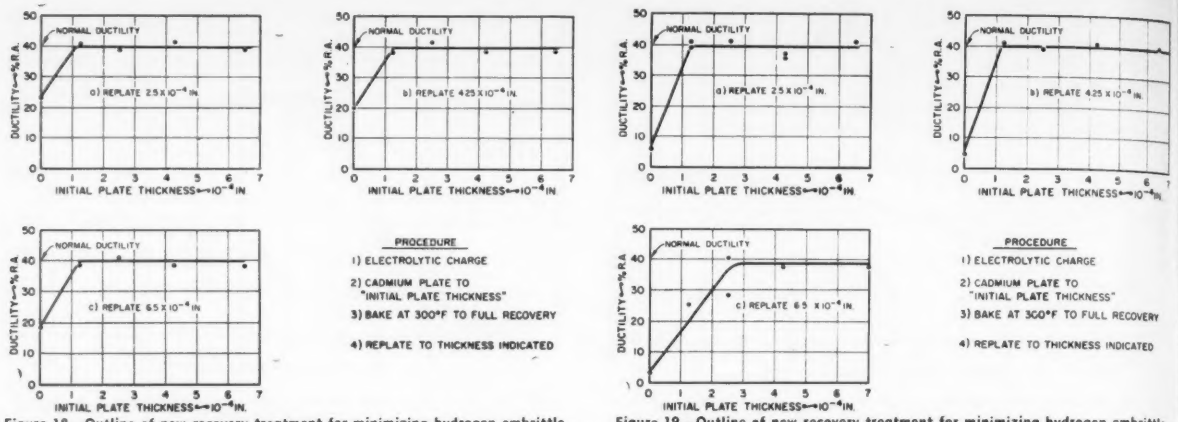
NORMAL DUCTILITY
a) REPLATE 2.5 X 10⁻⁴ IN.
b) REPLATE 4.25 X 10⁻⁴ IN.
c) REPLATE 6.5 X 10⁻⁴ IN.
DUCTILITY ⟷ % R.A.
INITIAL PLATE THICKNESS ⟷ 10⁻⁴ IN.
PROCEDURE
1) ELECTROLYTIC CHARGE
2) CADMIUM PLATE TO "INITIAL PLATE THICKNESS"
3) BAKE AT 300°F TO FULL RECOVERY
4) REPLATE TO THICKNESS INDICATED

Figure 18—Outline of new recovery treatment for minimizing hydrogen embrittlement. 230,000 psi strength level.

NORMAL DUCTILITY
a) REPLATE 2.5 X 10⁻⁴ IN.
b) REPLATE 4.25 X 10⁻⁴ IN.
c) REPLATE 6.5 X 10⁻⁴ IN.
DUCTILITY ⟷ % R.A.
INITIAL PLATE THICKNESS ⟷ 10⁻⁴ IN.
PROCEDURE
1) ELECTROLYTIC CHARGE
2) CADMIUM PLATE TO "INITIAL PLATE THICKNESS"
3) BAKE AT 300°F TO FULL RECOVERY
4) REPLATE TO THICKNESS INDICATED

Figure 19—Outline of new recovery treatment for minimizing hydrogen embrittlement. 270,000 psi strength level.

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Discussion by John P. Fraser, Shell Development Company, Emeryville, California:

The author stated that there is a finite "incubation period" before cracking begins, after which the crack grows continuously until rupture occurs. This sequence of events differs from that previously reported by Barnett and Troiano,[1] who reported initial crack propagation to be almost instantaneous through the outer layers of metal. I presume that the difference is due to the difference in distribution of hydrogen in the two cases. Barnett and Troiano stressed their specimens immediately after charging (very nonuniform distribution of hydrogen, with the concentration highest near the surface). In the present work, Troiano aged his specimens before stressing (distribution of hydrogen presumably nearly uniform across specimen). Is this interpretation correct? If so, what is the mechanism by which cracks initiate in a steel in which the hydrogen concentration is fairly low and uniform (i.e. steel was aged at elevated temperature after plating)?

In relation to the above, Bastien and Azou[2] found that hydrogen embrittlement was most marked in tensile tests on a carbon steel when speed of deformation caused failure in 10 seconds to 30 minutes. This suggests that diffusion of hydrogen to incipient cracks is rapid, which is consistent with the observations of other investigators. With this in mind, I wonder if the author would explain why he has used incubation periods of 50 to 60 minutes to crack initiation.

My other questions are rather brief:

1. Have plated metals other than cadmium been studied in connection with the delayed fracture problem?

2. What is the minimum thickness of cadmium needed to act as a barrier for hydrogen effusion?

3. Have any hydrogen analyses been made on specimens which have been charged with hydrogen and then aged at elevated temperatures? I would be particularly interested in the distribution of hydrogen after aging.

References

1. W. J. Barnett and A. R. Troiano. Crack Propagation in the Hydrogen-Induced Brittle Fracture of Steel. *Trans AIME*, 209, 486-494 (1957) April.
2. P. Bastien and P. Azou. Influence de l'amplitude et de la vitesse de déformation plastiques sur la ségrégation de l'hydrogene dans le fer et les aciers. *Comptes Rendus*, 232, 69-71 (1951) Jan. 3.

Reply by Alexander R. Troiano

Mr. Fraser's interpretation of the difference in incubation time reported here and in the earlier work is correct. In this later work, the hydrogen distribution is uniform across the specimen.

The incubation period is a function of the initial hydrogen concentration which accounts for the differences in incubation time for failure.

No plated metals other than cadmium have been studied. Minimum thickness of cadmium plate needed to act as a barrier is about 2 x 10^{-4} inch. However, this will depend upon the baking temperature. In high strength steels, hydrogen analyses do not show any difference between the plated and as-received material.

Discussion by Jerold Miles Van Orden, Lockheed Aircraft, Burbank, California:

Your test work on hydrogen embrittlement of steel has been performed mainly on small specimens and has evolved an embrittlement relief treatment. Should we expect the same degree of embrittlement relief on a much larger mass of metal with the same treatment?

With the hydrogen embrittlement treatments and special low embrittlement cadmium plating baths being developed, we should be able to produce practically non-embrittled parts. With subsequent service corrosion of the plate (with hydrogen evolution), will these parts become re-embrittled? Do you propose to study this phase of the problem?

Your reduction of area tests indicate freedom from embrittlement on specimens plated with a barrier coat (1 x 10^{-4} inches), relieved, then finish plated (6.5 x 10^{-4} inches). Can you explain why the barrier coat permitted relief and then prevented re-entry of hydrogen?

Reply by Alexander R. Troiano

We believe that it should be possible to obtain the same degree of relief in larger sections with the same treatment if the larger sections do not have more hydrogen in them prior to cleaning and plating. No study has been made of the possibility of embrittlement by service corrosion. In our opinion, this represents an interesting problem and a real possibility. As the cadmium plate becomes thinner by corrosion, the barrier effect will become less.

We believe that the barrier coat permits relief and then prevents re-entry because of the difference in temperature between the two operations. Relief is attained by baking at 300 to 400 F. Reentry would have to occur at lower plating bath temperatures. This, however, does point up the sensitivity of the method to the thickness of the first plate.

Any discussion of this article not published above will appear in the December, 1959 issue

Part 1—A Survey of the Literature

The Effect of Sigma Phase vs Chromium Carbides On The Intergranular Corrosion Of Type 316 and 316L Stainless Steel★

By DONALD WARREN

Abstract

A detailed survey of the literature on the intergranular corrosion of stainless steels is given to determine the relative effects of chromium carbides and sigma phase. The survey showed that at least eight different acids can cause intergranular corrosion of austenitic stainless steels if a continuous carbide network is present: acetic, hydrofluoric, hydrochloric, lactic, nitric, phosphoric, sulfurous and sulfuric acid. Only limited information was available on the effect of sigma phase on the intergranular corrosion resistance of stainless steels in these same acid environments. 3.2.2

Introduction

THE WELDING of austenitic stainless steels or their heat treatment in the 1000 to 1600 F temperature range causes formation of chromium carbides in the grain boundaries. A continuous grain boundary network of these carbides can lead to severe intergranular attack in certain corrosive environments. Three laboratory corrosion tests are commonly used to evaluate the susceptibility of austenitic stainless steels to intergranular attack due to carbide precipitation. These are the boiling 65 percent nitric acid test,(1) the acidified copper sulfate test and the 10 percent nitric-3 percent hydrofluoric acid test.

When certain grades of austenitic stainless steel such as Types 316, 316L and 321 are heated in the 1000 to 1800 F temperature range, another microstructural constituent known as sigma phase can form in the grain boundaries of the steel. This phase is an intermetallic compound consisting principally of iron and chromium and enriched in other ferrite-forming elements that may be present in the steel, such as Si, Mo, and Ti.

Of the three intergranular corrosion tests listed above, only the boiling 65 percent nitric acid test is sensitive to the presence of sigma phase in the steel. Even in the nitric acid test, the mere presence of sigma phase in the steel does not always mean a reduction in corrosion resistance. Instead, the form and distribution of the sigma phase are all-important in determining its effect on the nitric acid corrosion resistance.

Little is known about the effect of sigma phase on the resistance of stainless steel to those corrosive media commonly encountered in service. This, plus the fact that the nitric acid test is the only standard laboratory test seriously affected by sigma phase, has led several investigators[1,2,3] to question whether, in most environments, sigma phase is detrimental to the corrosion resistance of stainless steels.

The results of this investigation are reported in two parts: (1) a survey of the literature on the intergranular corrosion of austenitic stainless steels and its relation to the presence of chromium carbides and sigma phase and (2) a laboratory investigation of the relative effects of chromium carbides and sigma phase, as they are formed in Types 316 and 316L stainless steel, upon the intergranular corrosion resistance of these steels. Of greatest interest was the intergranular corrosion behavior in acid media other than nitric.

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(1) The electrolytic oxalic acid etching test is also used in conjunction with the nitric acid test to evaluate the intergranular corrosion resistance of certain grades of stainless steel.

About the Author

DONALD WARREN is research project engineer at the duPont Engineering Research Laboratory, Wilmington, Delaware. He has a BS in metallurgical engineering from the University of Kentucky and MS and PhD from Lehigh University. He joined the duPont staff in 1951.

Intergranular Corrosion as Related to Carbide Precipitation

Introduction

By 1932 it was already well known that austenitic stainless steels of the 18 Cr-8 Ni variety are subject to intergranular corrosion following exposure in the 1000 to 1600 F temperature range. This corrosion takes the form of preferential attack at the grain boundaries of the steel. If the intergranular attack is sufficiently severe, it can undermine the grains and eventually cause complete disintegration or powering of the metal.

During the years 1930-1933, numerous technical articles were published on intergranular corrosion.[4,5,6,7,8,9,10,11,12,13,14] Most of these early papers were concerned with (a) theories explaining the intergranular corrosion of stainless steels, (b) methods of testing stainless steel for susceptibility to intergranular attack and (c) means of preventing or minimizing intergranular corrosion in service. Such investigations as those of Strauss, Schottky and Hinnuber[14] and Aborn and Bain[4] showed that intergranular attack of austenitic stainless steels was associated with the precipitation of chromium carbides in the grain boundaries caused by exposure in the 1000 to 1600 F temperature range. Heat from welding was discovered to cause carbide precipitation in certain regions of the heat affected zone adjacent to the weld, thereby making these areas susceptible to intergranular attack.[7,15]

One of the earliest methods used to prevent intergranular corrosion was to reheat or anneal the welded steel at 1850 to 2000 F to dissolve the carbides, followed by rapid cooling. A second preventive method first used by the Krupp Works in Germany[5] was the addition of titanium to the steel. The titanium combined with the carbon, preventing the formation of damaging chromium carbides upon welding. The use of other alloying elements as carbide stabilizers, such as Nb and Ta, soon followed in this country.[5] As early as 1933, Bain, Rutherford and Aborn[5] showed that reducing the carbon content of austenitic stainless steels to less than about 0.03 percent would insure freedom from damaging carbide precipitation upon welding or short-time exposures (1 hr) to elevated temperatures. However, commercial alloys having such low carbon contents were not developed until after World War II.

The first explanation advanced to account for the intergranular corrosion of austenitic stainless steels was the chromium depletion theory of Strauss and others[14] and Aborn and Bain.[4,5] This theory postulated that the formation of chromium-rich carbides in the grain boundaries depleted the area adjacent to the grain boundaries in chromium and that the chromium-depleted areas were subject to preferential attack. The chro-

TABLE 1—Corrosive Service Environments in Which Austenitic Stainless Steels Have Undergone Intergranular Attack

Classification of Service Environment	Service Environment: Solution and Concentration	Service Environment: Temperature	Length of Service Exposure	Unit Undergoing Attack	Type of Stainless Steel	Condition of Steel Responsible for Intergranular Attack	Degree of Intergranular Attack	Reference
Acetic Acid	99.6% Acetic acid	245F	2 yr.	Condenser head	316	As cast	Severe I.G.A.[1] caused eventual replacement.	65, 66
	Acetic Acid (No conc. given)	....	1 yr.	Tank	316	Heated to red heat by plant fire	Gross I.G.A. leading to failure.	67
	55-75% Acetic acid +0.02-0.3% Salicylic acid	220F	....	Fractionating column	316	As-welded	I.G.A. in H.A.Z.[2] of welds caused complete penetration of column trays.	67
Nitric Acid	98% Nitric acid	Ambient	....	Welded flanges and fittings	304	As-welded	Marked I.G.A. in H.A.Z. of welds.	17
	Nitric acid (No conc. given)	....	....	Evaporator	302[3]	As-welded	Severe I.G.A. in H.A.Z. of welds.	15
	Soln. of silver nitrate and nitric acid	warm	....	Welded funnels and Dippers	302[3]	As-welded	I.G.A. in H.A.Z. of welds.	15
Phosphoric Acid	85% Phosphoric acid	350F (200 psi)	7 yr.	Polymerization vessels	317	Repair welded; no subsequent heat-treatment	Severe I.G.A. in H.A.Z. of repair welds leading to removal of vessel from service.	68, 69
	85% Phosphoric acid	....	....	Unit for Phosphoric acid manufacture	316	As-welded	I.G.A. in H.A.Z. of welds.	70
Sulfuric Acid	4% Sulfuric acid in methanol	Heated	1½ yr.	Steam-jacketed kettle	316	As-welded	Severe I.G.A. in H.A.Z. of weld causing failure.	18
Sulfuric Acid and Metallic Sulfates	Ferrous sulfate slurry in sulfuric acid	....	....	Centrifuge flights	316	Sensitized by hard-facing	Severe I.G.A. in region sensitized by hard facing.	17
	9% sulfuric acid + 7% ferrous sulfate	65F	21 days	Corrosion specimens	304	Welded and stress-relieved 2 hr. 1250F, A.C.	Severe I.G.A. causing very high corrosion rates (0.026 in./mo.).	30
	Sulfuric acid + copper sulfate	....	....	Support bars for pickling tank	302[3]	Hot-rolled	Severe I.G.A.	38
Hydrofluoric Acid and Ferric Sulfate	1.8-2.0% Hydrofluoric acid + 6-8% ferric sulfate	170F	15 min.	Pickled shells	302[3]	Sensitized by flame annealing	Band of severe I.G.A. and grain dropping around shell.	19
Mixed Sulfuric and Acetic Acids	0.5% Sulfuric acid + 0.3% acetic acid	150F	6 yr.	Agitators	316	One agitator: As-fabricated condition	Overall I.G.A. 1/16 in. deep.	17
						Another agitator: As-welded condition	Severe I.G.A. in H.A.Z. of weld.	
	0.5% Sulfuric acid + 28% acetic acid	120F	8 yr.	Agitator Hub	302[3]	As-cast	I.G.A. ¼ in. deep on all exposed surfaces.	17
Mixed Sulfuric and Nitric Acids	0.6% Sulfuric acid + 0.4% nitric acid	212F	....	.....	309	Spray-quenching insufficient for heavy sections	I.G.A. in heavier sections of equipment.	17
	Strong mixed acids (sulfuric acid + nitric acid)	....	2 yr.	Wringer basket	302	Inadequate (or no) heat-treatment	I.G.A. causing failure.	17
Mixed Lactic and Nitric Acids	50-85% Lactic acid + 10% nitric acid	175F	2½ yr.	Agitator	316	As-cast	I.G.A. leading to failure.	17
			2 yr.	Welded nozzle	317	As-welded	Severe I.G.A. in H.A.Z. of weld causing leakage.	17
Mixed Nitric and Hydrofluoric acids	10-12% Nitric acid + 1.5% hydrofluoric acid	140F	5-16 mo.	Pickling tanks	304 and 316	As-welded	Severe I.G.A. in H.A.Z. of welded joints causing failure.	26
Sulfurous Acid	Water, starch, + traces of sulfur dioxide	....	....	Centrifuge screen	302	Stress-relieved at 1150-1200F	I.G.A. 0.005 in. deep.	45
	Sulfite cooking liquor	....	75 days	Corrosion specimens	317	Sensitized at 950F and 1550F, slowly cooled	Heavy I.G.A.	45
	Sulfite solution	....	2 mo.	Elbows and bends in piping	302[3]	Sensitized by forming operation	Severe I.G.A. leading to metal disintegration.	71
	Sulfite digester acid (calcium bisulfite + 5-6% dissolved sulfur dioxide)	250F–320F (80 psi)	7-30 mo.	Sulfite digester fittings	302	As-cast	I.G.A. leading to removal from service.	72
Salts which Hydrolyze to Sulfuric Acid	Ammonium Sulfate Solution	....	....	Evaporator unit	317	As-welded	I.G.A. in H.A.Z. of welds.	73
	Impure water containing a large percentage of aluminum sulfate	....	Few months	Impeller casting	302[3]	Improper heat-treatment	Severe I.G.A. leading to removal from service.	15

[1] I.G.A. is an abbreviation for "intergranular attack."
[2] H.A.Z. is an abbreviation for "heat-affected zone."
[3] Those stainless steels which were not designated as to type or which were merely designated as "18-8" were assumed to be Type 302 stainless steel.

TABLE 1 (Continued)—Corrosive Service Environments in Which Austenitic Stainless Steels Have Undergone Intergranular Attack

Classification of Service Environment	Service Environment: Solution and Concentration	Service Environment: Temperature	Length of Service Exposure	Unit Undergoing Attack	Type of Stainless Steel	Condition of Steel Responsible for Intergranular Attack	Degree of Intergranular Attack.	Reference
Miscellaneous Environment....	Pickling acid	....	....	Wire pickling basket	304	As-welded	Severe I.G.A. in H.A.Z. of weld causing failure.	74
	Phenol + naphthenic acid	750F	10 yr.	Heater tubes	316	Sensitized by the 750F service temperature	Severe I.G.A., grain dropping and intergranular cracking leading to failure.	75
	Crude oil and its fractions	....	2 yr.	Fractionating tower	304	Stress-relieved at 1150-1200F	Severe I.G.A.	45
	Tree stumpage + extraction solvent for turpentine and rosin	....	6 mo.	Retort	302	As-welded	I.G.A. in H.A.Z. of welds.	45
	Sea Water	Warm	3 mo.	Shaft	302	Heat-treated at 1200F by mistake	Severe I.G.A. causing complete penetration of shaft at keyway.	41

[1] I.G.A. is an abbreviation for "intergranular attack."
[2] H.A.Z. is an abbreviation for "heat-affected zone."
[3] Those stainless steels which were not designated as to type or which were merely designated as "18-8" were assumed to be Type 302 stainless steel.

mium depletion theory still remains the most popular explanation for intergranular attack in austenitic stainless steels. However, this theory does not fit all of the experimentally observed facts concerning intergranular corrosion of stainless steels. Consequently, other theories have been suggested. One of the simplest of these, first advanced by Kinzel,[4] is that selective corrosion at the grain boundaries is due to the strain and electrolytic effect (galvanic action) associated with the actual presence of chromium carbides in the grain boundaries.

Service Environments Causing Intergranular Corrosion of Austenitic Stainless Steels

Although numerous technical papers on intergranular corrosion were written in the early thirties, few detailed descriptions were given of actual service failures of austenitic stainless steel due to intergranular attack. For example, Aborn and Bain[4] state that "almost any sort of acid corrosion will proceed to develop the exaggerated intergranular attack," but they fail to cite any of these acids or to provide other details. They point out that the effect of intergranular attack is more pronounced if there is "some moderate oxidizing agent present" to develop protection of the grain faces against corrosion. The scarcity of information concerning intergranular corrosion failures still existed as late as 1949 when the American Society for Testing Materials (ASTM) sponsored a "Symposium on Evaluation Tests for Stainless Steels."[16] At that symposium, Brown, DeLong and Myers[17] commented that "probably the weakest point in our present knowledge of intergranular corrosion and our use of evaluation methods is lack of information on exactly which of the many and widely varied service environments for which stainless steels are employed will attack susceptible material intergranularly."

For these reasons, this literature survey was made. Table 1, which summarizes the results of the survey, is a list of those service environments in which severe intergranular attack of austenitic stainless steels has been observed.

The data in Table 1 show that intergranular corrosion failures occurred in service environments involving at least seven common acids: acetic, hydrofluoric, lactic, nitric, phosphoric, sulfuric and sulfurous. Intergranular corrosion failures have also occurred in such miscellaneous environments as warm sea water and in crude oil plus its fractions. A recent publication[18] on the application of stainless steel in the chemical industries indicates that the following environments can also cause severe intergranular corrosion of stainless steel: chlorinated solvents, solutions acidified with hydrochloric acid (e.g., starch slurry) and pickle liquors or sauces (these primarily cause intergranular pitting where carbides are present).

Most of the intergranular corrosion failures listed in Table 1 occurred as a result of welding stainless steel containing more than 0.03 percent C without subsequent annealing. However, some failures were due to stress-relieving heat-treatments, elevated service temperatures or inadequate final annealing treatments.

The number of service failures of austenitic stainless steel due to intergranular corrosion has been greatly reduced over the past ten years. This reduction has largely resulted from improved fabrication and heat-treatment practices and use of the low carbon (0.03 percent max.) or stabilized grades of stainless steel for service in the as-welded condition. Occasional intergranular corrosion failures of austenitic stainless steel still occur, however.[19]

Laboratory Test Solutions Causing Intergranular Corrosion of Austenitic Stainless Steels

Tables 2 and 3 list those laboratory test solutions in which intergranular corrosion of austenitic stainless steels has been observed. Test solutions used by only one or two investigators are included in Table 2; the more widely used test solutions are listed in Table 3. It should not be inferred that these test environments will always produce intergranular attack either in the laboratory or in service.

All seven of the common acids which intergranularly attacked stainless steel in service (see Table 1) also produced intergranular corrosion in laboratory tests (see Tables 2 and 3). In addition to these seven acids, Streicher[20] found that inhibited solutions of boiling 10 percent formic, sulfuric and oxalic acids caused intergranular corrosion of Type 304 stainless steel sensitized for one hour at 1250 F. Laboratory tests also showed that ferric chloride solutions can produce severe intergranular attack on sensitized stainless steel.[7,21] A boiling 10 percent chromic acid solution is unusual in that it causes severe intergranular corrosion of austenitic stainless steel in the annealed as well as the sensitized condition.[21]

Table 3 shows that the three corrosion tests[(2)] most commonly used to determine the susceptibility of austenitic stainless steels to intergranular attack are (1) the boiling 65 percent nitric acid test, (2) the acidified copper sulfate test and (3) the nitric-hydrofluoric acid test. Only the first two of these tests have been standardized by the ASTM.[23,24,25] The boiling 65 percent nitric acid test has found its widest use in this country; the acidified copper sulfate test has been more widely applied in Europe. Comparison of the three tests listed in Table 3 is available in the ASTM "Symposium on Evaluation Tests for Stainless Steels."[16,17] Application of these three tests to welded samples has been reviewed by Bloom and Carruthers[26] and Thielsch.[27] Each of the three tests is briefly discussed below as to origin, application, and limitations.

Boiling 65 Percent Nitric Acid Test. This test, commonly called the Huey test after its founder,[28] was originally developed to determine the general corrosion resistance of stainless steels to

[(2)] The electrolytic oxalic acid etching test was not included in Table 3 because it is not an actual corrosion test. However, this test is widely used to evaluate the susceptibility of austenitic stainless steels to intergranular corrosion.[22]

TABLE 2—Laboratory Test Solutions in Which Austenitic Stainless Steels Have Undergone Intergranular Attack

Type of Solution	Test Environment: Solution and Concentration	Test Environment: Temp.	Length of Test	Type of Steel Tested[1]	Condition of Sample[2]	Method Used to Evaluate I.G.A.[3]: Appearance	Bending	Weight Loss	Micro. Exam.	Remarks	Reference
Acetic Acid.....	99.5% Acetic Acid	Boiling	Two 48-hr. Periods	304	H.T.	X		X	X	Mild I.G.A.	32
Chromic Acid....	10% Chromic Acid	Boiling	110 hr.	304, 316	H.T.	X		X	X	Produces severe I.G.A. on both annealed and sensitized stainless steel	21
Ferric Chloride	5% Ferric Chloride	Boiling	.....	316	H.T.	X				Severe I.G.A. and disintergration	21
	25% Ferric Chloride	Cold	17 hr.	302	W.	X				I.G.A. and sugaring in H.A.Z.[4] of weld	7
Formic Acid.....	10% Formic Acid (Inhibited)[5]	Boiling	2400 hr.	304	H.T.	X		X		Light grain dropping No increase in in corrosion rate	20
Lactic Acid	50% Lactic Acid	Boiling	Five 48-hr. Periods	304	H.T.	X		X		I.G.A. leading to high corrosion rate	76
	85% Lactic Acid	Boiling	Two 48-hr. Periods	304	H.T.	X		X	X	Definite I.G.A. with increased corrosion rate	32
Nitric Acid	12.5% Nitric Acid	Boiling	96 hr.	302	H.T.	X		X		Severe I.G.A. and sugaring of surface	41
	55% Nitric Acid	Boiling	Three 48-hr. Periods	304	H.T.			X		I.G.A. resulting in increased corrosion rate	1
	70% Nitric Acid	210°F	One Hundred 24-hr. Periods	302, 304	H.T.	X		X		Severe I.G.A. caused large increase in corrosion rate	71
Nitric and Hydrochloric Acids	6% Nitric + 0.9% Hydrochloric Acid (Inhibitor Added)	90-140F	17 hr.	302	H.T.	X	X			Severe I.G.A. reflected in bend test	7
Oxalic Acid	10% Oxalic Acid (Inhibited)[5]	Boiling	1500 hr.	304	H.T.	X		X		Severe I.G.A. and grain dropping. Large increase in corrosion rate	20
Phosphoric Acid	60%, 70%, and 85% Phosphoric Acid	Boiling	Two 48-hr. Periods	304, 316	H.T.	X		X	X	I.G.A. varied in degree, but never caused a large increase in corrosion rate	32
Sodium Bisulfate	10% Sodium Bisulfate (Inhibited)[5]	Boiling	2400 hr.	304	H.T.	X		X		I.G.A. and grain dropping. Small increase in corrosion rate	20
Sulfamic Acid	10% Sulfamic Acid (Inhibited)[5]	Boiling	2400 hr.	304	H.T.	X		X		I.G.A. and grain dropping. Appreciable increase in corrosion rate	20
Sulfuric Acid	0.1% Sulfuric Acid + 5-10% Ferric Sulfate	Boiling	20-170 hr.	302	H.T.	X	X		X	Severe I.G.A. on steels containing 0.12% C	11, 41
	0.5% Sulfuric Acid	Boiling	24-48 hr.	302, 316	H.T., W.	X	X			Severe I.G.A.	3, 77
	1% Sulfuric Acid	Boiling	.....	302, 316	H.T., W.	X	X			I.G.A. tended to be masked by general surface attack	77
	3% Sulfuric Acid	Boiling	15 hr.	316	H.T.	X		X		Severe I.G.A. and grain dropping	21
	10% Sulfuric Acid (Air-Free)	158F	Two 48-hr. Periods	316, 317	H.T., W.	X	X	X		Definite I.G.A. leading to cracking upon bending	33, 58
	10% Sulfuric Acid	176F	24 hr.	Worthite[6]	H.T.	X				Severe I.G.A. and grain dropping	64
	10% Sulfuric Acid	Boiling	3 hr.	302, 304	H.T.	X		X	X	Severe I.G.A. and extensive grain dropping	8
	10% Sulfuric Acid (Inhibited)[5]	Boiling	1500 hr.	304	H.T.	X		X		Severe I.G.A. and grain dropping Large increase in corrosion rate	20
Sulfurous Acid	Saturated Solution of Sulfurous Acid	Cold	2-3 days	302	H.T.	X		X		Severe I.G.A. with complete penetration of sample	78

[1] Where the stainless steel was described only as 18-8, it was assumed to be Type 302.
[2] Sample condition is designated either "H.T." for heat-treated or "W." for welded. Heat treatments varied from 1-1000 hr. at temperatures from 1000°-1400°F.
[3] I.G.A. is used as an abbreviation for intergranular attack.
[4] H.A.Z. is used as an abbreviation for heat-affected zone.
[5] Inhibited by the presence of ferric ions.
[6] Worthite is a 20% Cr-24% Ni-3% Mo-2% Cu-3% Si alloy.

boiling 65 percent nitric acid. In the article which first introduced the test,[28] Huey merely implied that the test was influenced by microstructural changes in the steel caused by heat-treatment. No data were given to support this implication.

Later, as related by Brown,[29] it was learned that the test could be used to detect improperly heat-treated material that would be subject to severe intergranular attack in nitric acid service. Thus, at first, the nitric acid test was used as a simulated-service test. Then, as intergranular corrosion of improperly heat-treated stainless steel was observed in service environments other than nitric acid, the boiling 65 percent nitric acid test was used to detect possible susceptibility to intergranular attack in these other environments as well.

The nitric acid testing procedure as originally described by Huey was the basis of an ASTM standard adopted in 1944.[24] There has been little or no variation in the test procedure used by various investigators.[17,30,31,32,33,34,35,36] The nitric acid test has always been a quantitative one based on the weight loss occurring in five 48-hour periods. Ellinger and Bibber[37] have correlated the weight-loss measurements in the nitric acid test with electrical resistance data and microscopic measurements of the depth of intergranular attack.

The boiling 65 percent nitric acid test has often been criticized,[1,30,32,35] but still remains one of the most widely used methods for evaluating the susceptibility of stainless steel to intergranular corrosion. Most of the criticism has stemmed from either a misunderstanding as to the purpose of the test or the fact that the test is sensitive to the presence of phases other than carbides in certain grades of stainless steel, which is the case with Types 316L and 321.

Acidified Copper Sulfate Test. The origin of the acidified copper sulfate test is a striking example of how chance laboratory or field observations often develop into items of major technical significance. The acidified copper sulfate test grew out of an investigation by W. H. Hatfield[38] of an intergranular corrosion failure at a silver works in Sheffield, England. The failure was that of an austenitic stainless steel bar used for supporting articles in a pickling tank. The pickling solution was chemically analyzed, and laboratory corrosion tests were made in individual components of the solution. These tests revealed that sulfuric acid and copper sulfate were collectively responsible for the intergranular corrosion of the supporting bar (the copper sulfate was a corrosion product produced by the pickling of nickel silver parts). The mixture formed by pickling nickel silver articles in a commercial sulfuric acid pickling solution became the basis of a widely used test for susceptibility to intergranular corrosion.

The acidified copper sulfate test is often referred to as the Strauss test.[39] According to Payson,[9] Strauss was the first to suggest the use of the test in this country and consequently received most of the credit for its origination. However, Hatfield's claim[38] to the origination of the acidified copper sulfate test is supported by others.[12,40] Strauss states[14] that he learned of the test through an early edition of Monypenny's book[41] as translated into German by Schafer in 1928. Strauss modified the original Hatfield solution into the 7:10 mixture[(3)] (10 percent copper sulfate (hydrated)-10 percent sulfuric acid by weight) listed in Table 3.

Shortly after its introduction in this country, the acidified copper sulfate test was again modified to a 1:8 mixture by Rutherford and Aborn.[13] They claimed that the reduction in copper sulfate content increased the severity of intergranular attack obtained with the test. This claim was later disputed by Rosenberg and Darr,[42] who stated that the copper sulfate content in Aborn's 1:8 solution was too low to prevent excessive general attack on many of the steels tested. As a result, Rosenberg and Darr used a 3:8 mixture which was subsequently used by others[17,33,43,36] and has now been tentatively proposed by ASTM as a "Recommended Practice for Conducting the Acidified Copper Sulfate Test for Intergranular Attack in Austenitic Stainless Steels."[25] Previously, another ASTM specification[23] and the ASME Code[44] have specified use of the 1:8 mixture.

Originally, the only criteria used to evaluate stainless steel specimens exposed to the acidified copper sulfate test were the presence of cracks after bending and the ring test, in which the corroded specimen is dropped on a hard surface of wood or stone. Rutherford and Aborn[13] were among the first to use electrical resistance measurements for evaluating the degree of intergranular corrosion produced by the acidified copper sulfate test. A more detailed discussion of evaluation methods for the acidified copper sulfate test has been given by Ebling and Scheil.[45]

The principal limitations to the acidified copper sulfate test are as follows: (1) long test times (500 to 1000 hr.) are required to develop maximum sensitivity to damaging carbide precipitation[16] and (2) a quantitative evaluation requires electrical resistance measurements which are costly and time-consuming and which limit the dimensions of the specimen that can be used.

Recent research by Rocha[46] indicates that the rate of intergranular attack in an acidified copper sulfate solution can be greatly increased by placing the stainless steel test specimen in contact with copper chips. It is claimed that the testing time can be reduced from 200 hours to 24 to 48 hours by using this technique.[46]

Nitric-Hydrofluoric Acid Test. The origin of the nitric-hydrofluoric acid test is not at all clear. As far as can be determined, Becket and Franks[47] and Payson[48] first reported the use of a nitric-hydrofluoric acid solution for testing stainless steel. These investigators used the mixed acid solution to reveal carbide precipitation in the heat-affected zones of welded samples of austenitic stainless steel.

According to Franks,[49] in 1929 it was found at Union Carbide and Carbon Research Laboratories, and at others, that a 158 F (70 C) solution containing 1 to 3 percent hydrofluoric acid and about 10 percent nitric acid satisfactorily descaled the annealed 18 Cr-8 Ni stainless steels. In a series of experiments, this solution was used to descale specimens of stainless steel which had been deliberately sensitized for laboratory test purposes. The sensitized specimens completely disintegrated after a very short time due to intergranular attack by the nitric-hydrofluoric acid pickling solution. Because of its high sensitivity to precipitated carbides, the nitric-hydrofluoric acid solution was then used on welded samples to determine if the heat-affected zones were subject to intergranular corrosion.

Later investigators[17,31,33,36,50,51,52,53] have employed solutions varying in concentration from 10 to 15 percent nitric and 2 to 4 percent hydrofluoric acid at temperatures ranging from 60 C to boiling (see Table 3). However, at the time of the ASTM "Symposium on Evaluation Tests for Stainless Steels,"[16] use of a 10 percent nitric-3 percent hydrofluoric acid solution at 158 to 176 F (70 to 80 C) seemed to have become standard practice. In most cases, specimens subjected to the nitric-hydrofluoric acid test were evaluated by appearance and weight loss; in one case, electrical resistance measurements were used.[50]

A nitric-hydrofluoric acid solution is more potent than any other test solution in revealing susceptibility to weld decay (intergranular attack on the heat-affected zones of welded stainless steel).[16] Despite its potency, the nitric-hydrofluoric acid test has never been used on a quantitative basis for evaluation of stainless steel principally because it produces severe general corrosion on annealed material. The high general corrosion rates tend to obscure the weight losses caused by intergranular attack, making quantitative evaluation by weight-loss measurements difficult.

Recent work[54] indicates that the nitric-hydrofluoric acid test can be quantitatively applied to Type 316L stainless steel. The ratio of the corrosion rate for the sensitized condition to the corrosion rate for the annealed condition is used for evaluation rather than the absolute corrosion rate for the sensitized condition. A ratio of approximately one showed that the sensitized specimen had corroded at the same rate as the annealed specimen and that no intergranular corrosion had occurred. Where the sensitized specimen underwent serious intergranular attack and excessive grain-dropping, the ratio was generally 2.0 or more.

Intergranular Corrosion as Related To Sigma Phase

Little is known about the influence of sigma phase on the corrosion resistance

(3) The ratio 7:10 was obtained by dividing the weight percentage of $CuSO_4$ (dehydrated) by the weight percentage of H_2SO_4.

TABLE 3—Standard Laboratory Corrosion Tests Used to Determine the Susceptibility of Austenitic Stainless Steels to Intergranular Corrosion

Title of Test	Test Conditions: Solution and Concentration	Temp.	Length of Test	Method Used to Evaluate I.G.A.[1]: Appearance	Ring Test	Bend Test	Electrical Resistance	Weight Loss	Micro. Exam.	Remarks	Reference
Standard Nitric Acid Test[2]	65% Nitric Acid	Boiling	Five 48-hr. Periods					X		Standard A.S.T.M. test procedure	24
	65% Nitric Acid	Boiling	Five 48-hr. Periods	X			X	X	X		37
Acidified Copper Sulfate Test	Hatfield's Solution (3:4 Mixture)[3] 111 g. $CuSO_4 \cdot 5H_2O$ (6.1%) 98 g. Conc. H_2SO_4 (8.1%) Water to make one liter	Boiling	One 72-hr. Period	X	X	X				Original acidified copper sulfate test	38, 40, 77
		Boiling	One 72-hr. Period	X		X		X	X	Studied effect of copper sulfate concentration	12
		Boiling	1-56 days	X			X				10
	Aborn's Solution (1:8 Mixture)[3] 13 g. $CuSO_4 \cdot 5H_2O$ (0.8%) 47 ml. Conc. H_2SO_4 (7.9%) Water to make one liter	Boiling	One 72-hr. Period	X	X	X				A.S.T.M. and A.S.M.E. Code test procedure	23, 44
		Boiling	Up to 1500 hr.	X		X			X		26, 34, 35, 79
		Boiling	Up to 1000 hr.	X			X				5, 45 13, 37
	New Standard Solution (3:8 Mixture)[3] 100 g. $CuSO_4 \cdot 5H_2O$ (5.4%) 100 ml. Conc. H_2SO_4 (15.5%) Water to make one liter	Boiling	One 72-hr. Period	X		X				New A.S.T.M. test Procedure	25
		Boiling	Up to 300 hr.	X		X					33, 43
		Boiling	Up to 2000 hr.	X	X	X	X		X		17, 36, 42
		Boiling	Up to 1300 hr.	X	X	X					6, 9, 11 14, 71, 78
	Strauss or Krupp Solution (7:10 Mixture)[3] 10% (100 g.) $CuSO_4 \cdot 5H_2O$ (7.1%) 10% (100 g.) Conc. H_2SO_4 (9.6%) 80% (800 ml.) Water	Boiling	48 hr.	X	X	X	X	X	X		50, 80
		Boiling	Up to 1000 hr.	X					X		8, 79
Nitric-Hydrofluoric Acid Test	10% Nitric-2% Hydrofluoric Acid	140F	Forty-eight ½-hr. Periods	X						Welded samples only	53
	10% Nitric-3% Hydrofluoric Acid	163F	Four ½-hr. Periods	X				X			17
		158F	Five 1-hr. Periods	X				X			33, 36
		176F	Three 4-hr. Periods	X				X			26, 51
	10% Nitric-4% Hydrofluoric Acid	140F	Four ½-hr. Periods	X						Welded samples only	52
	15% Nitric-3% Hydrofluoric Acid	Hot	One 2-hr. Period	X			X	X	X		50
		Boiling	Two 4-hr. Periods	X						Welded samples only	52
	Nitric-Hydrofluoric Acid (No concentrations given)	.....	One 2-hr. Period	X						First recorded use of this test (welded samples only)	47, 48

[1] I.G.A. is used as an abbreviation for intergranular attack.

[2] The boiling 65% nitric acid test was standardized in 1933 only three years after its introduction. Therefore, reference is made only to the standard A.S.T.M. testing procedure. Reference (37) was included because it was the only one where electrical resistance measurements were used in conjunction with the nitric acid test.

[3] The ratios given for each mixture are the ratios of the copper sulfate content to the sulfuric acid content in weight per cent.

of stainless steels.[55,56,57] However, it has been established that sigma phase can cause accelerated corrosion of stainless steel in boiling 65 percent nitric acid and in hot, concentrated (40 to 80 percent) sulfuric acid solutions. Aside from these media, there has been no evidence to indicate that the presence of sigma phase is necessarily damaging to the corrosion resistance. Moreover, there are no records of field corrosion failures that can be directly attributed to sigma phase.

Effect of Sigma Phase on Standard Laboratory Corrosion Tests

Of the three laboratory intergranular corrosion tests previously discussed, only the boiling 65 percent nitric acid test is sensitive to the presence of sigma phase. In the case of the standard nitric acid test, the form and distribution of the sigma phase in the steel determine its effect on the corrosion resistance. For example, sigma phase in the form of an envelope of extremely fine particles around the grains will cause severe intergranular corrosion to occur in boiling 65 percent nitric acid,[33,56] but when the sigma phase is coalesced in the form of isolated pools in the grain boundaries, it has no effect on the nitric acid corrosion resistance.[58,55]

Most of the experimental evidence concerning the effect of sigma phase on the nitric acid corrosion resistance is based on the behavior of Type 316L stainless steel.[17,58,33,34] A number of in-

vestigators[17,33,34] have shown that if Type 316L stainless steel is sensitized 1 to 2 hours at 1200 to 1400 F, it undergoes severe intergranular corrosion in the boiling 65 percent nitric acid but is not affected by either an acidified copper sulfate solution or a 10 percent nitric-3 percent hydrofluoric acid solution. The latter two testing solutions are capable of producing severe intergranular attack if carbides are present. Consequently, the nitric acid attack must have been caused by some other intergranular constituent such as sigma phase.[33,34]

In the studies described above, Binder and Brown[33] were able to establish the presence of sigma phase by X-ray diffraction techniques. They heat-treated a specimen of very low carbon (0.006 percent) Type 316L stainless steel for 10 minutes at 1380 F. In this condition, although its micro structure showed no evidence of grain-boundary phases, the steel underwent severe intergranular attack in boiling 65 percent nitric acid. Despite the lack of microscopic evidence of phase changes, residues that were electrolytically isolated from the steel contained sigma phase (according to X-ray diffraction patterns of the residue). This same steel after heat-treatment for 24 hours at 1380 F contained clearly defined particles of sigma phase in the grain boundaries. In this form, the sigma phase had little or no effect on the nitric acid corrosion resistance. Franks, Binder and Bishop[58] were among the first to recognize that, if the sigma phase in Type 316L stainless steel is coagulated by heat-treatment at 1600 F, it no longer has a detrimental effect on the nitric acid corrosion resistance. Many others[21,33,34,59,60] have verified this observation.

Cooke[61] and Shirley[62] have shown that, in the case of an 18Cr-8Ni-3Mo-1Ti steel, boiling 65 percent nitric acid preferentially attacks ferrite which has been partially transformed to sigma phase. However, with complete transformation of the ferrite to sigma phase, the nitric acid attack is greatly reduced. Bloom and Carruthers[26] have made similar observations regarding ferrite in Type 316L stainless steel weld metal. They found that in the as-welded condition such ferrite is selectively attacked in boiling 65 percent nitric acid but not in acidified copper sulfate or in 10 percent nitric-3 percent hydrofluoric acid solutions. It was postulated, therefore, that partial transformation of the ferrite to sigma phase during the welding cycle was responsible for the nitric acid attack.

Effect of Sigma Phase on Media Other Than Standard Laboratory Corrosion Tests

The only corrosive environment, other than nitric acid, which has been reported in the literature as having attacked sigma phase in austenitic stainless steels is 25 percent sulfuric acid at 40 C. Smith and Bowen,[63] and later Cooke,[61] showed that when sigma was present in an 18Cr-8Ni-3Mo-1Ti steel, the corrosion rate in 25 percent sulfuric acid at 40 C was twelve times that for the annealed condition. In this case, approximately half of the original ferrite content (40 percent) had been transformed to sigma phase by heat-treatment for 4 hours at 1380 F).

The same investigators observed that in less corrosive media, such as 5 percent sulfuric acid at 40 C or acidified boiling ammonium sulfate solution, there was no decrease in corrosion resistance due to sigma phase. Others[64] have likewise pointed out that sigma phase may seriously increase the corrosive attack in hot, strong (40 to 80 percent) sulfuric acid solutions but that it is generally not harmful in more dilute (1 to 5 percent) solutions of hot sulfuric acid.

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Any discussion of this article not published above will appear in the December, 1959 issue

Technical Practices Committee Members Named

Maitland and Battle Appointed Chairmen; New Members Listed

T. J. Maitland of American Telephone & Telegraph Co., New York, N. Y., has been appointed chairman of the Technical Practices Committee, succeeding E. C. Greco, United Gas Corp., Shreveport, La. Jack L. Battle of Humble Oil & Refining Co., Houston, has been appointed vice chairman.

Chairmen of Group Committee 1, 3, 5, 7 and 9, who are members of the Technical Practices Committee, have been appointed also.

Mr. Maitland has been active in NACE since 1944 and has written several technical papers presented at national conferences and published in CORROSION. He is chairman of Technical Unit Committee T-2B on Anodes for Impressed Currents, Unit Committee T-4B on Corrosion of Cable Sheaths and also Task Group T-4B-1 on Lead and Other Metallic Sheaths.

He is electrolysis and protection engineer with his company's Long Line Department. A graduate of Worcester Polytechnic Institute in electrical engineering and former faculty member of the University of New Hampshire, he has devoted his career to corrosion control, cable construction and maintenance methods of long distance telephone cables.

Vice Chairman Battle, who succeeds Mr. Maitland in that office, is supervising chemical engineer in Humble's Production Department. He has been with Humble since 1933. In 1952 he helped organize Unit Committee T-1H on Oil String Casing Corrosion. He has served as chairman of Group Committee T-1 for the past two years, the Oil and Gas Production Symposium at the 1958 and 1959 Conferences and 1958 South Central Region Meeting. He is also a member of eight other NACE committees.

An active member of NACE since 1944, Mr. Battle is also a member of the American Petroleum Institute, Mining and Metal Engineers and the American Chemical Society. Several of his technical papers have been published in CORROSION and other technical publications.

New Group Committee Chairmen

New chairmen of Technical Group Committees 1, 3, 5, 7 and 9 have been appointed and become members of the Technical Practices Committee:

T-1 on Corrosion in Oil and Gas Well Equipment: W. C. Koger, Cities Service Oil Co., Bartlesville, Okla., succeeds Jack L. Battle, who has been T-1 chairman for two years. After graduating from the University of Arkansas with a BS in chemical engineering in 1944, Mr. Koger joined Cities Service, where he is head of the Corrosion and Oil Treating Section of the Oil Production Division. An active NACE member since 1951, he helped organize the West Kansas Area T-1H Task Group, has been chairman of Unit Committee T-1H and has been vice chairman of Group Committee T-1 for the past two years.

Maitland

Battle

Mercer

Koger

Dillon

T-3 on General Corrosion: Dale A. Vaughan, who succeeds A. H. Roebuck, is affiliated with Battelle Memorial Institute, Columbus, Ohio. An active NACE member since 1944, he is a graduate of Ohio State University with majors in physics and math. His work in corrosion has been the application of X-ray and electron diffraction methods to the study of corrosion products and passivating films.

T-5 on Corrosion Problems in the Process Industries: C. P. Dillon, Union Carbide Chemicals Co., Charleston, W. Va., succeeds R. I. Zimmerer as chairman. An active NACE member since 1950, Mr. Dillon is chairman of Unit Committee T-5A and a member of several other committees. He has a BS in chemistry from Georgetown University and holds membership in ACS and ASM.

T-7 Corrosion Coordinating Committee: C. L. Mercer of Southwestern Bell Telephone, Houston, who succeeds C. A. Erickson as chairman, is one of the original members of NACE and currently is chairman of Committee T-7D, South Central Region Corrosion Coordinating Committee and is vice chairman of Task Group T-4B-5 on Non-Metallic Sheaths and Coatings.

T-9 on Marine Biological Deterioration: Albert P. Richards, president of William F. Clapp Laboratories, Inc., Duxburg, Mass., who succeeds C. M. Wakeman as T-9 chairman, has been a NACE member for two years. A graduate of the University of Massachusetts, he has been in marine deterioration research and consulting work for over 15 years. He holds membership in several professional organizations and societies in addition to NACE.

Group Committee T-1 Hears Unit Reports Given at New Orleans

Technical Group Committee T-1 on Corrosion of Oil and Gas Well Equipment heard reports from its unit committees at the New Orleans meeting last October. Summary of the reports is given below.

T-1A

Unit Committee T-1A on Corrosion of Oil and Gas Well Equipment, Los Angeles Area reported that two meetings were held since the 1958 Conference in San Francisco. At the April meeting, internal casing corrosion in the Los Angeles Basin was discussed; at the June meeting an inspection tour of the water injection facilities in the Long Beach Harbor area was held. Also, a general discussion of corrosion in waterflooding operations was held, and a paper was presented on the subject of bacteriological methods used in evaluating corrosion and plugging problems in waterfloods.

T-1B

Unit Committee T-1B on Condensate Well Corrosion is primarily concerned with completion and corrosion control

(Continued on Page 72)

New Committee Planned On Marine Deterioration

A committee under Technical Group T-9 on Marine Biological Deterioration is being considered and will be concerned with barrier coatings and their application.

Persons interested in joining this committee should contact Roger H. Hopkins, Roger Hopkins Company, 15 North Euclid, Pasadena, Cal.

Gackenbach

Fenner

Roebuck

NEW CHAIRMEN for Unit Committees T-5D on Plastic Materials of Construction and T-3B on Corrosion Products have been elected. T-5D chairman is R. E. Gackenbach of American Cyanamid Co., New York, N. Y. T-5D vice chairman is Otto H. Fenner of Monsanto Chemical Co., St. Louis, Mo. Chairman of T-3B is A. H. Roebuck, Western Co., Midland, Texas, who was re-elected. T-3B vice chairman is Dale A. Vaughan of Battelle Memorial Institute, Columbus, Ohio.

TECHNICAL REPORTS

on

HIGH PURITY WATER CORROSION

Symposium on Corrosion by High Purity Water by Committee T-3F on High Purity Water which includes:

Introduction to Symposium on Corrosion by High-Purity Water by John F. Eckel.

Corrosion of Structural Materials in High-Purity Water by A. H. Roebuck, C. R. Breder and S. Greenberg.

Corrosion Engineering Problems in High-Purity Water by D. J. DePaul.

The Importance of High-Purity Water Data to Industrial Applications by W. Z. Friend. Per Copy.....$1.50

Symposium on Corrosion by High Purity Water. Five Contributions to the Work of NACE Technical Committee T-3F on High Purity Water. Pub. 57-22.

Measurement of Corrosion Products in High Temperature, High Pressure Water Systems by A. S. Sugalski and S. I. Williams.

Corrosion of Aluminum-Nickel Type Alloys in High Temperature Aqueous Service by F. H. Krenz.

Corrosion of Aluminum in High Purity Water by R. J. Lobsinger and J. M. Atwood.

The Storage of High Purity Water by Richard R. Diesk.

Water Conditions for High Pressure Boilers by D. E. Voyles and E. C. Fiss. Per Copy.....$1.50

Remittances must accompany all orders for literature the aggregate cost of which is less than $5. Orders of value greater than $5 will be invoiced if requested. Send orders to National Association of Corrosion Engineers, 1061 M & M Bldg., Houston, Texas. Add 65c per package to the prices given above for Book Post Registry to all addresses outside the United States, Canada and Mexico.

NATIONAL ASSOCIATION OF CORROSION ENGINEERS

1061 M & M Bldg. Houston 2, Texas

19th Committee Joins Coordinating Group

The Toledo and Northwestern Ohio Committee on Corrosion becomes the 19th committee to affiliate with NACE's Group Committee T-7 Corrosion Coordinating Committee and the third committee to affiliate directly with Unit Committee T-7B North Central Region Corrosion Coordinating Committee.

The Ohio committee officers are Chairman L. H. West, associated with Standard Oil Co., Fostoria, Ohio, Vice Chairman O. J. Mauter of Ohio Bell Telephone Co., Toledo, and Secretary I. H. Heitkamp, associated with Toledo Edison Co.

The committee voted to affiliate with T-7B at its January meeting in Toledo.

The Ohio committee functions as a clearing house for exchange of engineering information on corrosion problems arising in the territory of Toledo and northwestern Ohio. Three regular meetings are held each year in January, April and October with special meetings called when required.

West

Mauter

Heitkamp

Unit Committee T-1B Hears T-1B-1 Task Group Report

Technical Unit Committee T-1B on Condensate Well Corrosion discussed Task Group T-1B-1's report (Well Completion and Corrosion Control of High Pressure Gas Wells) which has been published in the February issue of CORROSION. The task group plans to meet this year to discuss any new information concerned with high pressure wells. If no new information and no need to continue the task group are presented, the group plans to recommend to T-1B that the task group be abandoned.

The problem of emulsion in gas condensate systems was discussed also at the T-1B meeting held in New Orleans.

J. A. Caldwell Elected Vice Chairman of T-1

Caldwell

J. A. Caldwell, senior research engineer with Humble Oil & Refining Co., Houston, has been elected vice chairman of Group Committee T-1 on Corrosion in Oil and Gas Well Equipment. He succeeds W. C. Koger, who becomes committee chairman.

Mr. Caldwell has a MS in physics from Louisiana State University and has been an active NACE member since 1946. He has been chairman of Unit Committee T-1D, chairman of the Houston Section and is presently chairman of the South Central Region.

Group Committee T-1—

(Continued From Page 71)

of high pressure wells through the T-1B-1 Task Group, which prepared a report presented at the Oil and Gas Symposium at the South Central Region Meeting in New Orleans.

T-1C

T-1C Unit Committee on Sweet Oil Corrosion presented a report of its meeting concerned with the use of inhibitor squeezes. The report classified squeeze treatment into three types: short, medium and long. Between 250 and 300 wells were reported to have been squeeze-treated by 12 companies. Twenty-four of these wells were considered failures in regards to corrosion control; the remainder were considered successful, a percentage of 91. The success or failure of a well was based on iron counts, caliper surveys and/or failures.

T-1D

Unit Committee T-1D has three task groups: T-1D-2 West Texas-New Mexico, T-1D-3 Western Kansas and T-1D-4 on Oilfield Pumps. During the past six months, activities have centered around a study by the West Texas-New Mexico Task Group covering inhibitors, pump corrosion, sucker rods and tubing, plastic pipe and tanks, storage tanks and treaters. A questionnaire was sent to 100 operators with 20 operators mailing returns covering 8992 wells, of which 2971 were considered corrosive. Cathodic protection was being used to protect about 62 percent of the heater treaters.

T-1K

Unit Committee T-1K on Inhibitors for Oil and Gas Wells gave a report from its Task Group T-1K-1 (Testing inhibitors for sour crude use) that a second edition of a standard screening test for sour crude inhibitors has been prepared and distributed.

T-1K is considering the formation of a new task group on new methods of approach, the subject of film persistency and other items not covered by other task groups of the committee.

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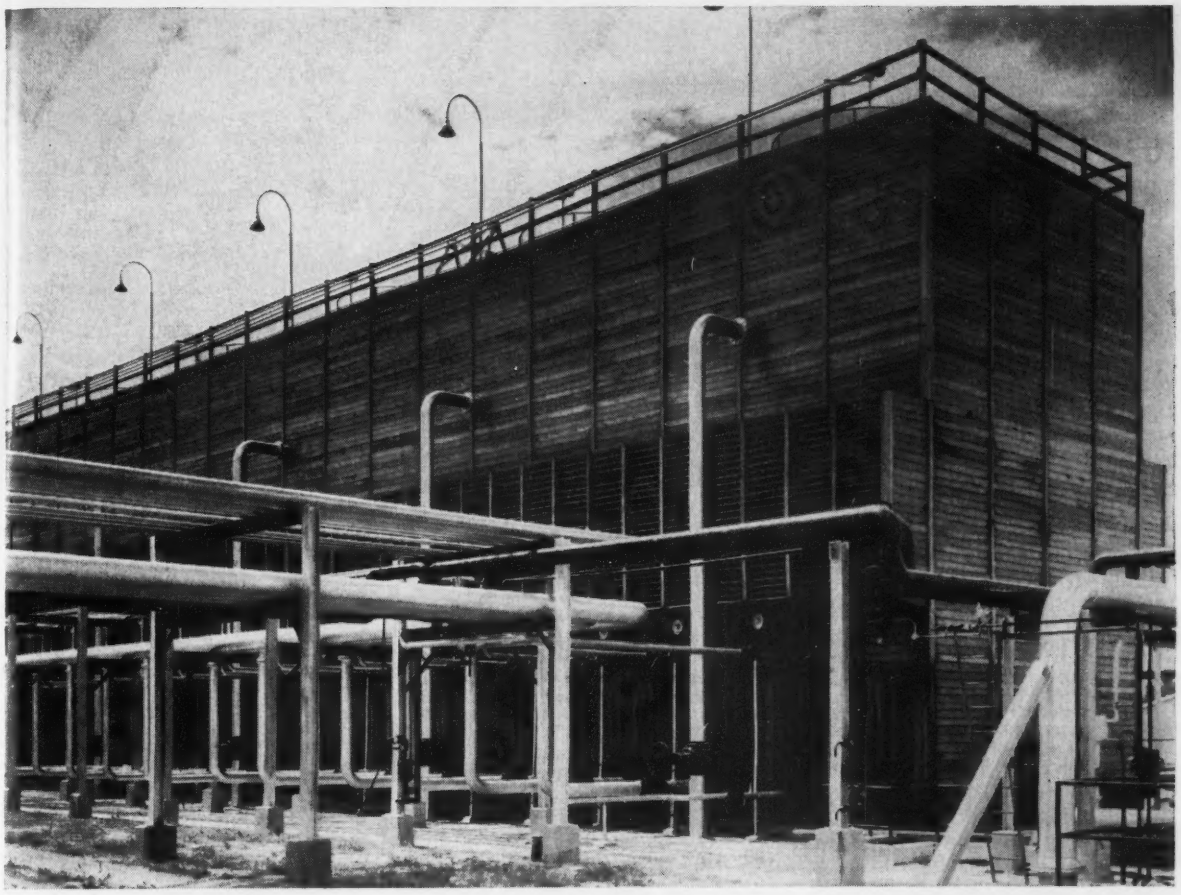
Deadline for news stories in CORROSION is the tenth of the month.



CHROMATE COMPOUNDS STOP CORROSION *before it starts!*

Long before the first drop of water contacts metal in any large recirculating water system, steps should be taken to prevent corrosion.

Early in the design stage, plan on adding a chromate inhibiting compound to recirculating water—from the moment operation starts. That way, clean metal stays clean. Chromate inhibiting compounds will also arrest corrosion in older systems that have been operating without protection, even where rust and scale make it more difficult for inhibitors to reach and protect the metal.

Chromates work two ways: they make waters non-corrosive while making metal surfaces corrosion-resistant. Chromate inhibiting compounds are easy and inexpensive to use. Since conditions and equipment vary, a variety of compounds are available to meet individual requirements.

For the names of manufacturers of corrosion inhibiting compounds containing Mutual Chromium Chemicals, or information on Mutual Chromium Chemicals, mail the coupon below.

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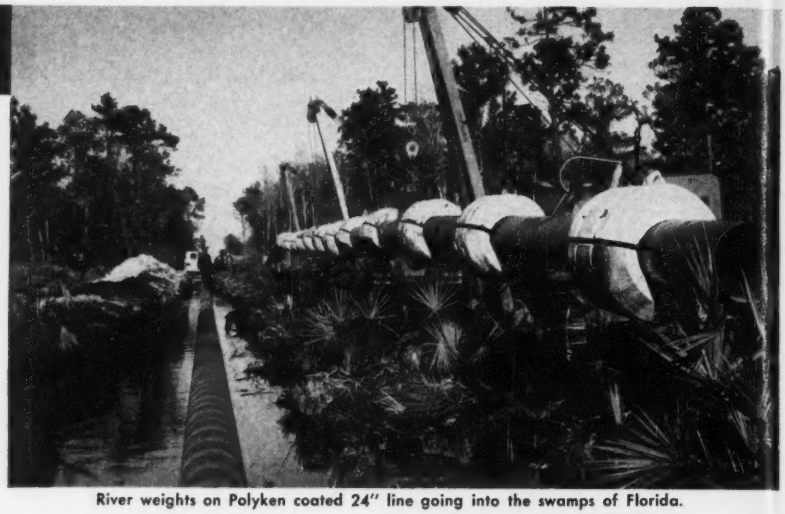
11-4

Cathodic Protection

CPS Undertakes Largest SINGLE PACKAGED Pipe Line Protection Job



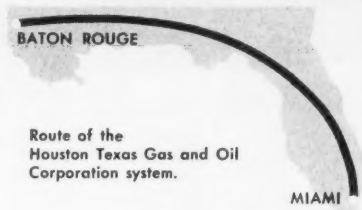
River weights on Polyken coated 24" line going into the swamps of Florida.

Houston Texas Gas and Oil Corporation selected CATHODIC PROTECTION SERVICE to design and install a complete cathodic protection system for their new natural gas pipeline. This line runs from Baton Rouge, Louisiana, to Miami, Florida, covering a distance of 1616 miles.

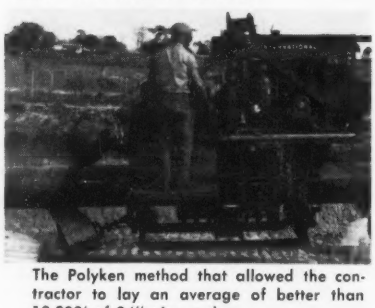
This is the largest *single packaged* pipeline cathodic protection job yet undertaken. CPS is in a unique position to handle just such a job . . . serving as a central source . . . for all phases of corrosion mitigation on any new pipeline.

The Maloney type-57 Insulator chosen for the Houston Texas Gas and Oil Corporation pipeline.

CPS has its own engineering crews who obtain the information necessary to complete the detailed design of a cathodic protection system. Plus this, CPS furnishes all materials, labor, equipment and engineering supervision to install cathodic protection on a *packaged* basis. Because it is a



package, CPS can immediately follow the laying contractor, providing complete protection to the pipeline system soon after the last foot of pipe is laid. In addition to the cathodic protection package, CPS also serves as a central source of supply for other materials which were used on this pipeline, i.e., Polyken polyethylene tape, Maloney type 57 plastic casing insulators and the L & M pipeline markers, casing vents and test lead boxes.



The Polyken method that allowed the contractor to lay an average of better than 10,000' of 24" pipe a day.

This represents a package within a package which CPS can deliver: (1) L & M gas line marker (2) Gas vent with cover (3) Test lead box with lead wires.

We at CPS believe a strong relationship is essential among manufacturer, supplier and customer. We know through experience that ties of this sort mean more initial and repeat business. If you are considering a similar big pipeline job, why not talk it over with us and see how our *single packaged* cathodic protection can give you cost-cutting advantages. It will cost you nothing to find out.

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TULSA 4407 S. Peoria RIverside 2-7393

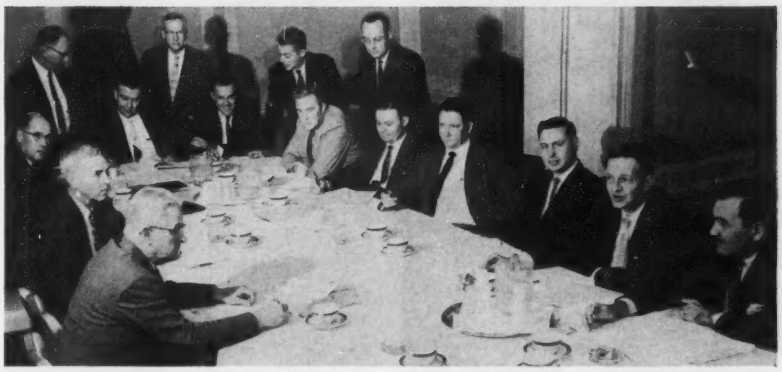
Appalachian Short Course Date Set for June 2-4

Course to Be Given In 64 Classes and Field Demonstrations

Sixty-four classes and field demonstrations have been scheduled for the Fourth Annual Appalachian Underground Corrosion Short Course to be held June 2-4 at West Virginia University in Morgantown.

Hugh P. Godard, new NACE president affiliated with Aluminum Laboratories Ltd., Kingston, Ontario, Canada, will give one of the opening addresses on June 2.

Program for the short course is given below.



PROGRAM COMMITTEE of the Appalachian Underground Corrosion Short Course are shown above planning course sessions set for June 2-4 at West Virginia University in Morgantown. They are from left to right: Charles W. Beggs, Public Service Electric & Gas Co., Newark, N. J., C. L. Brown, U. S. Beureau of Mines, Pittsburgh, Pa., Art Erickson, Peoples Natural Gas Co., Pittsburgh, Pa., Charles Rutter, Equitable Gas Co., Pittsburg, Pa., Ed Benson, New York Telephone Co., Buffalo, N. Y., K. E. Sawyer, Western Union Telegraph Co., Boston, Floyd G. Cole, Orange & Rockland Utilities, Inc., Middleton, N. Y., P. L. Knott, Dresser Industries, Bradford, Pa., James L. Mills, United Natural Gas Co., Oil City, Pa., Hugh W. Hetzer, Union Carbide Chemicals Co., South Charleston, W. Va., Frank B. Burns, Kerr-McGee Oil Co., Wynnewood, Okla., Charles Stoneking, Hope Natural Gas Co., Clarksburg, W. Va., William Cook, Jr., Pipe Line Service Corp., Glenwillard, Pa., Herb Byrns, Columbia Gas Service Corp., Columbus, Ohio, and Frank E. Costanzo, Manufacturers Light & Heat Co., Pittsburgh, Pa. Program chairman is Charles Rutter.

Tuesday Afternoon, June 2

12:00—1:30
Exhibits and Luncheon

1:30—3:00
Fundamentals of Corrosion: G. C. Cox consultant
Field Practices: L. H. West, Standard Oil Co.
Some Fundamentals of Lead Cable Corrosion: K. E. Sawyer, Western Union Telegraph Co.
Expendable Anodes: T. Lennox, Federated Metals Division, American Smelting and Refining Co.
Corrosion Resistance of Cast Iron Pipe: D. W. Johnson, Cast Iron Pipe Research Association.
Asphalt Coatings: A. J. Hoiberg, Flintkote Company.
Corrosion Testing Instruments: M. C. Miller, instrument manufacturer.
Polyvinyl Chloride Pipe: A. H. Henning, General Tire & Rubber Co.

3:15—4:45
Fundamentals of Corrosion: W. A. Koehler, West Virginia University.
Fundamentals of Plant Testing for Underground Corrosion: A. W. Peabody, Ebasco Services, Inc.
Instrumentation and Equipment: A. L. Ayres, New Jersey Bell Telephone Co.
Expendable Anode Applications: M. A. Riordan, Rio Engineering Co.
Carbonate Deposits for Pipe Protection: R. F. McCauley, Michigan State University.
Use of Wax Coating for Protecting Underground Pipe: R. Moehl, Trenton Corporation.
Corrosion Resisting Characteristics of Aluminum Alloys: M. E. Carlisle, Aluminum Company of America.

6:00—9:00
Exhibits and Movies at Field House

Wednesday Morning, June 3

8:30—10:00
Mud Tub Demonstration: C. A. Erickson, Jr., Peoples Natural Gas Co.
Construction Practices: W. R. Curley, Royston Laboratories, Inc.
Fundamentals of a Corrosion Survey: E. K. Benson, New York Telephone Company.
Pipe Type Cables: W. A. Sinclair, Detroit Edison Company.
Plastic Coated Pipe: H. M. McDaniel, Republic Steel Corp.
A New Concept in Recording Voltmeters: W. H. Bollinger, Electrical Instruments.
Cathodic Protection of Casings in Gas Storage Wells: W. R. Lambert, United Fuel Gas Co., and G. Campbell, West Virginia University.

10:15—11:45
Electrochemical Principles: C. M. Rutter, Jr., Equitable Gas Co.
Protection of Gas Service Lines in Non-Stray Current Areas: P. P. Skule, East Ohio Gas Co.
Interpretation of Survey Data in a Stray Current Area: E. G. Carlson, Bell Telephone Company of Pennsylvania.
Rectifier Installations and Protection: L. O. McCormick, Baltimore Gas & Electric Co.
Water Pipeline—Exterior Corrosion Control: H. R. Ludeker, Electro Rust-Proofing Corp.
Hot Applied Tapes: J. B. Casey, Tapecoat Company.
Instruments Used in Soil Resistivity Measurements: L. M. Bull, Manufacturers Light & Heat Co.
Weather Versus Cathodic Protection: F. E. Costanzo, Manufacturers Light & Heat Co.

Wednesday Afternoon, June 3

12:00—1:30
Exhibits and Luncheon

1:30—3:00
Fundamental Analysis of Stray Current Corrosion: P. Hort, Atlantic Pipe Line Co.
Demonstration of a Theory of Cathodic Protection: H. F. Byrns and W. F. Taylor, Columbia Gas System Service Corp.
Interpretation of Survey Data in the Non-Stray and Current Area: R. W. Ackerman, American Telephone and Telegraph Company.
Rectifier Units for Cathodic Protection: B. Husock, Harco Corp.
Corrosion and Its Control in Public Water Systems: L. Haynes, West Virginia Water Service Corp.
Cold Applied Tapes: L. L. Whiteneck, Plicoflex, Inc.
Electrical Holiday Detectors: J. P. Rasor, Tinker and Rasor.
Microbiological Corrosion of Underground Structures: F. E. Kulman, Consolidated Edison Company of New York.

3:15—4:45
Electrolysis Current Related to a Distribution System: G. J. Powers, Equitable Gas Co.
Application of Cathodic Protection: D. A. Tefankjian, Texas Eastern Transmission Corp.
Electrolysis Currents Related to a Power Cable System: D. W. Kissinger, Duquesne Light Company.
Practical Interference Testing: R. L. Seifert, Tennessee Gas Pipeline Co.
Elements of Water Transmission Corrosion: W. L. LaDue, Bureau of Water Supply, Department of Public Service
Coal Tar Epoxy Coatings: N. T. Shideler, Pittsburgh Coke & Chemical Co. Earth Current Meter: F. E. Costanzo, Manufacturers Light & Heat Co.
A Report on the Effectivenesss of Armored Gaskets in Couplings for Cathodic Protection of Gas Distribution Lines: G. D. Kish, Dresser Industries.

6:00—9:00
Exhibits and Motion Pictures

7:00
General Committee Dinner Meeting

Thursday, Morning, June 4

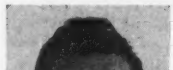
8:30—10:00
Soil Bacterial Corrosion: Roger E. McVey, Manufacturers Light & Heat Co.
Intermediate Analysis of Stray Current Corrosion: T. S. Watson, Socony Mobil Oil Co., Inc.
Corrosion Surveys and Mitigative Measures of Power Cables in Non-Stray Current Areas: Harry Neugold, Jr., Electro Rust-Proofing Corp.
Design and Installation of Cathodic Protection: N. P. Peifer, Manufacturers Light & Heat Co.
Cold Water Corrosion of Copper—Related Problems: M. E. Flentje, American Water Works Service Co., Inc.
Review of Properties of Coal Tar Coatings: W. F. Fair, Jr., Koppers Company, Inc.

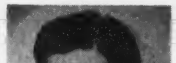
(Continued on Page 77)

April Issue to Cover Chicago Conference

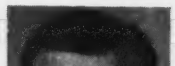
Reports of the 15th Annual NACE Conference in Chicago will be published in the April issue of CORROSION. The conference was being held as this issue went to press.

1960 Dallas Conference Chairmen Appointed


Spalding


Meek

Hodgdon


Catlow

McDowell

Mathay

Coburn

Priest

Robbins

McCarthy

Shultz

Norris

Dallas Conference Plans for 1960 Are Making Progress

Plans are well advanced for the 16th NACE Conference to be held in Dallas, March 14-18, the first annual conference in the Southwest in eight years. The 1960 Corrosion Show will be given concurrently with the conference.

All conference meetings will be held in the Dallas Memorial Auditorium on two floors above the exhibit area where the 1960 Corrosion Show is scheduled.

J. C. Spalding, Jr., Sun Oil Co., Dallas, is chairman of the General Conference Committee. Vice chairman is J. Gordon Meek, Metal Goods Corp., Dallas.

Co-chairmen of the Technical Program Committee are S. K. Coburn, Association of American Railroads, Chicago, and David K. Priest, Pfaudler Co., Rochester, N. Y.

Other chairmen are S. D. Hodgdon, Metal Goods Corp., Dallas, exhibits; T. E. Catlow, Galloway's Pipe Guard, Houston, publicity; K. W. Robbins, Otis Engineering Corp., Dallas, registration; and R. A. McCarthy, Hill, Hubbell and Co., Dallas, entertainment.

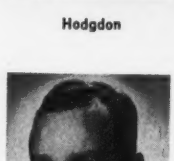
Mdms. R. A. McCarthy and J. C. Spalding, Jr., both of Dallas, will be in charge of the Ladies' Program.

Chairmen for the 16 symposia schedule for the 1960 Conference are listed below:

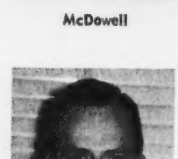
Cathodic Protection: H. C. Boone, Peoples Light, Gas and Coke Co., Chicago.

Chemical Industries: L. W. Gleekman, Wyandotte Chemical Corp., Wyandotte, Mich.

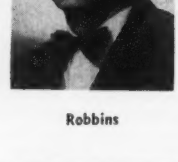
(Continued on Page 77)


Boone

Gleekman

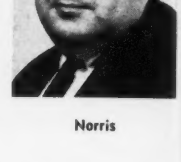

Koger

Jeffares


Ida

Freedman

Kittredge


Gackenbach

Quimby

English

Janssen

Mercer

Plans for 1960 Corrosion Show Are Given

Advance information on the 1960 Corrosion Show to be held in Dallas March 15-17 concurrently with the 16th NACE Conference is available by writing R. W. Huff, Jr., NACE Exhibits Manager, 1061 M & M Bldg., Houston 2, Texas. Advance folders on the Corrosion Show were distributed at the Chicago Conference last month. The 1960 show will be the first national NACE exhibition held in the Southwest since the 1952 meeting at Galveston.

The advance folders give pertinent information for exhibitors and contains a floor plan of the 115 booths available. The show will be held in the Dallas Memorial Auditorium, which offers many advantages to exhibitors including easy accessibility to the exhibit area from the meeting rooms which are in the same building. The auditorium is fairly new with ample parking and loading space.

All booths will be 8 x 10 feet with necessary connections for water, drainage, electricity and air. Booths will be arranged to give best traffic flow to as many booths as possible.

Meeting rooms for the technical sessions of the annual conference will be on the two floors above the exhibit area. Stairs at both ends of the exhibit area lead to these rooms.

The number of booths available for the 1960 Corrosion Show is smaller than the number available for previous NACE shows, according to Mr. Huff, so those interested in the exhibit should make reservations early.

Dallas Conference—

(Continued From Page 76)

Corrosion in the Missile Industry: Noble Ida, Martin Co., Denver, Colo.

Corrosion Inhibitors: A. J. Freedman, Standard Oil Co., of Indiana, Whiting, Ind.

Corrosion Principles: W. S. Quimby, Texas Co., Beacon, N. Y.

Educational Lectures: J. L. English, Oak Ridge National Laboratories, Oak Ridge, Tenn.

Elevated Temperature: D. W. McDowell, International Nickel Co., New York, N. Y.

General Corrosion: W. L. Mathay, United States Steel Corp., Monroeville, Pa.

High Purity Water: B. G. Schultz: Westinghouse Atomic Power Division, Pittsburgh, Pa.

Marine: R. O. Norris, Cities Service Research and Development Co., Lake Charles, La.

Oil and Gas: W. C. Koger, Cities Service Oil Co., Bartlesville, Okla.

Pipe Line—General: G. M. Jeffares, Plantation Pipe Line Co., Atlanta, Ga.

Plastics: J. B. Kittridge, Duriron Co., Inc., Dayton, Ohio.

Protective Coatings: R. E. Gackenbach, American Cyanamid Co., New York, N. Y.

Refining Industry: W. Janssen, American Oil Co., Texas City, Texas.

Utilities Industries: C. L. Mercer, Southwestern Bell Telephone, Houston.

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1959 Western Region Conference will be held Sept. 29-Oct. 1 at Bakersfield, California, at Bakersfield Inn.

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Southeast Region's 1959 meeting will be held in Jacksonville, Fla., Oct. 1-2.

Missile Industry Corrosion To Be New Symposium Topic

Corrosion in the Missile Industry is a new symposium included among the 16 to be given at the 16th Annual Conference in Dallas.

Other symposia are on cathodic protection, corrosion inhibitors, protective coatings, corrosion principles, general corrosion, corrosion at elevated temperatures and corrosion problems in the chemical, marine, oil, gas, plastic, utilities, pipe line and refining industries.

Co-chairmen in charge of the 1960 Technical Program are S. K. Coburn, Association of American Railroads, Chicago, and David K. Priest, Pfaudler Co., Rochester, N. Y.

'61 and '62 Dates Are Set For Annual Conferences

Dates and locations for two more annual conferences have been set.

The 17th Annual NACE Conference and 1961 Corrosion Show will be held March 13-17, 1961, at the Hotel Statler in Buffalo, N.Y.

The 18th Annual Conference and 1962 Corrosion Show have been set for March 18-22, 1962, at the Municipal Auditorium in Kansas City, Mo.

Appalachian Course—

(Continued From Page 75)

Pipe Locators: J. H. Reynolds and E. E. Gross, Norfolk Redevelopment and Housing Authority.

New Coupling Developments to Speed Field Joining and Protection of Pipe: G. D. Kish, Dresser Industires.

10:15—11:45

General Corrosion Control Practices: J. A. Schauers, Atlantic Pipe Line Company.

Safe Operating Pratcies Related to Electrical Current Flows Upon Natural Gas Pipe Line Structures: I. E. Davis, Ohio Fuel Gas Co.

Bonding and Ground (Power): A. S. Brooker, Public Service Electric & Gas Co.

Duties of a Coating Inspector: John B. Vrable, New York State Natural Gas Co.

Reference Electrodes: T. J. Maitland, American Telephone and Telegraph Co.

Corrosion Problems in the Chemical and Metallurgical Industries: W. W. Hodge, Mellon Institute of Industrial Research.

Wrought Iron—Its Production, Properties and Performance: H. K. Seifers, Jr., A. M. Byers Company.

Thursday Afternoon, June 4

12:00—1:30

Exhibits

1:30—3:45

Field Domonstrations: Soil resistivity, pipe to soil potentials, surface potentials, line current measurements, magnesium anode installation, cathodic protection with rectifier, other underground structures and summary of demonstration.

•

The 1959 North Central Region Meeting will be Oct. 20-22 in Cleveland, Ohio.

COMPLETE CORROSION SERVICE . . .

Wayne A. Johnson, president, and his associates in Corrosion Rectifying Co., Inc., are well-known corrosion prevention specialists with headquarters in Houston and Southeastern Divisional offices in New Orleans. Their work is concentrated on designing and installing corrosion rectifying systems for pipelines, refineries and oil producing companies. Call on them for prompt and experienced surveys, systems and materials.

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South Central Region News

Burnett

Fugazzi

Kent

Hopkins

Patterson

Mossholder

Sinton

Wheatlake

Goodrich

Lewis

Hartkemeier

McKinnel

Becker

Chairmen Appointed For 1960 Conference Meeting in Denver

Committee chairmen have been selected for the 1959 South Central Region Conference to be held in Denver, Colo., Oct., 12-16 at the Cosmopolitan Hotel. Tentative schedule for the conference also has been set.

Chairmen of the conference committees are as follows:

General Arrangements: G. E. Burnett, Bureau of Reclamation, Denver Federal Center, Denver.

Local Arrangements: J. F. Fugazzi, Public Service Company of Colorado, Denver.

Housing Subcommittee: Bryan Patterson, Hill-Hubbell Company, Denver.

Entertainment Subcommittee: Steve Mossholder, Dresser Manufacturing Co., Denver.

Transportation Subcommittee: B. K. Wheatlake, manufacturer's representative, Denver.

Ladies Program Subcommittee: Mrs. Frances L. Goodrich, Denver.

Tours Subcommittee: P. W. Lewis, Bureau of Reclamation, Denver Federal Center, Denver.

Technical Program Committee: Chairman Leonard W. Hartkemeier, Colorado School of Mines, Golden, and Vice Chairman William P. McKinnell, Jr., Ohio Oil Co., Littleton, Colo.

Properties and Projection Subcommittee: Gale W. Hodgkin, Armco Drainage and Metal Products, Inc., Denver.

Finance Committee: Henry K. Becker, Wyco Pipeline Co., Denver.

Registration Subcommittee: Edward L. Kent, Jr., Kohler-McLister Paint Co., Denver.

Publicity Committee: John R. Hopkins, Protecto Wrap Co., Denver.

Printing Subcommittee: M. H. Sinton, Colorado Interstate Gas Co., Aurora, Colo.

Conference Schedule

Tentative schedule for the conference is as follows:

Technical Committee Meetings: October 12, 13 and 14.

Symposia meetings: October 14 and 15.

Tour of Bureau of Reclamation Engineering Laboratories: October 16.

Social and Business Events: Trustee Luncheon, October 13; Fellowship Hour, October 13; Authors' Breakfast, October 14; Dinner-Dance, October 14; and annual Luncheon, October 15.

Technical Program

Four symposia are scheduled with two others being planned in addition to two educational lectures. The four symposia already set are Corrosion in Oil and Gas Production, Corrosion in the Pipe Line Industry, Corrosion in Fresh Water and Corrosion in the Utilities and Communication Industry.

Plans are being made to include symposia on Corrosion in the Chemical Process and Refinery Industries and High Temperature Corrosion in the Atomic Age, according to Mr. Hartkemeier, chairman of the technical program.

•

Permian Basin Section heard Floyd Blount of Magnolia Petroleum Company speak on non-destructive testing of oil field tubular goods at the February meeting.

•

Corpus Christi Section held a special joint meeting with the Corpus Engineering Societies in February. Guest speaker at the banquet meeting was Edwin Vennard, managing director of the Edison Electric Institute, New York City.

•

Shreveport Section had M. J. Oliver, Arkansas Fuel Oil Corp., as speaker at the March meeting. He spoke on cathodic protection maintenance.

The section's April 7 meeting will be in the Caddo Hotel.

•

North Texas Section held a panel discussion at its March meeting on the subject "Cathodic Protection for the Oil Producing Industry." Moderator was J. C. Spalding, Jr., Sun Oil Co., Dallas. Guest panelists were Jim Landers of Continental Oil Co., Ponca City, Okla., and M. D. Folzenlogen of Atlantic Refining Co., Dallas.

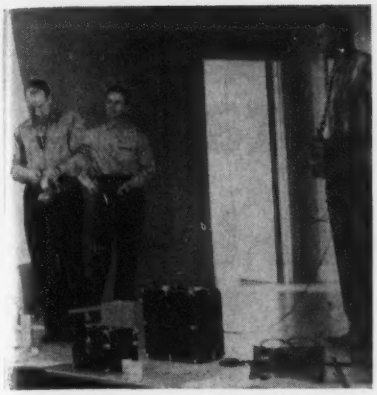
At the section's February meeting, William H. Wade of the University of Texas spoke on radiation effects on corrosion.

•

Teche Section guest speaker for the March meeting was M. A. Riordan of Rio Engineering, Houston. Harry Waldrip of Gulf Oil spoke at the February meeting on paints and epoxy resins.

•

East Texas Section heard M. J. Olive, Arkansas Fuel Oil Corp., speak at the February meeting on "Application of Cathodic Protection in the Oil Field." He discussed various types of installations and maintenance of cathodic protection systems.



TULSA CORROSION SHORT COURSE registrants saw this demonstration of the use of the Pearson holiday detector during the February 11-13 tenth annual course given at Mayo Hotel, Tulsa. W. E. Huddleston, Engineering Co., (left) is shown pointing out some of the features of the foot electrode worn by members of the team using the Pearson device. Registration for the course was over 215.

Western Region News

San Francisco Bay Area Section will hold its April 8 meeting at Spenger's Fish Grotto, 1919 Fourth St., Berkeley, Cal. Aaron Wachter of Shell Development Corp., Emeryville, Cal., will discuss volatile corrosion inhibitors. The discussion will include various inhibitor types, how they work, their suitablity, specific applications and their future possibilities.

Section officers for 1959 installed at the February meeting are President-Chairman John P. Fraser of Shell Development Corp., Emeryville, Cal., Vice Chairman Roy O. Dean of Pacific Gas & Electric Co., Emeryville, and Secretary-Treasurer John W. Caldwell of the Bunker Hill Co., San Francisco.

•

Portland Section heard Robert S. Baynham of Dearborn Chemical Company speak on treatment of boilers at the February meeting. R. V. Moorman, past section chairman affiliated with Bonneville Power Administration, Portland, was appointed chairman of a special committee to consider a corrosion control short course to be held in Portland in the near future.

Frank Nisbett of Shell Oil Co., Anacortes, Washington, spoke on "Organization and Functions of a Corrosion Control Department" at the March meeting.

•

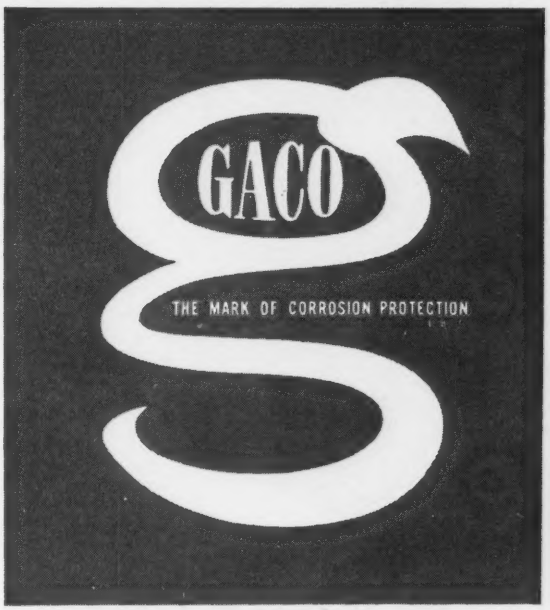
The Western Region Conference is scheduled for September 29-30, October 1, 1959, at Bakersfield, Cal.

•

NACE regional and sectional secretaries who do not have a supply already may get copies of the form "News Report for Corrosion" from Central Office on request. The form helps in providing a complete news story concerning meetings. The news deadline for CORROSION is the tenth of the month preceding month of issue.

NEW
GACO N-700A
Neoprene Maintenance Coating

Provides lasting protection against corrosive fumes, splash and weathering, yet requires no acceleration or heat cure! Improved formulation of performance-proven GACO liquid neoprene has better brushability and color stability, can be brush applied to a thickness of 3½ mils in a single coat or hot sprayed, and coats over old or new neoprene without lifting the under coating. Write for details on how "lower applied cost" GACO N-700A can save you maintenance dollars.



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Challenge: When paint or ordinary corrosion protection products won't do the job, specify GACO. A complete line: Neoprene, Natural Rubber, Vinyls, Liquids, Sheet, Putties . . . performance-proven in applications throughout the world. *And A Complete Service.* There's a GACO Corrosion Specialist in your area prepared to serve your needs. Write for further information—we'll forward case studies of interest.

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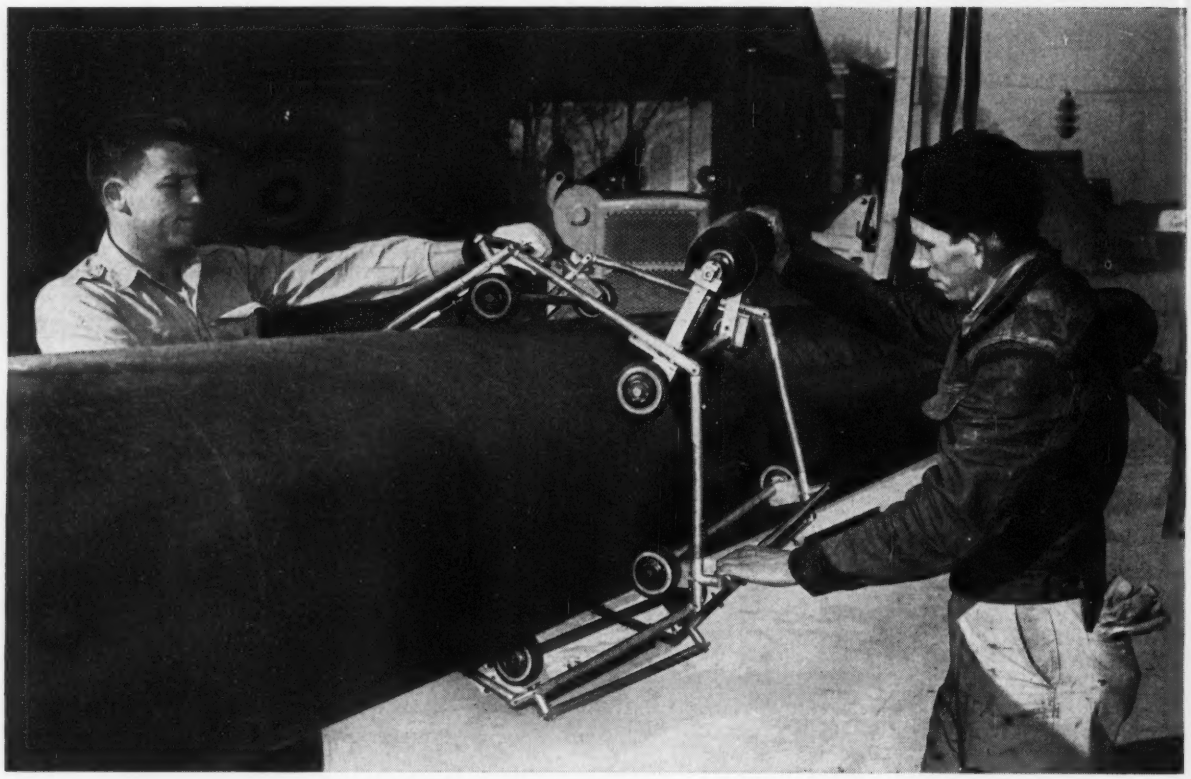

Upper New York State utility company turns to Polyken tape to get the job done fast... and sure... in congested areas

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Allen Cathodic Protection Company, Inc.

Houston, Texas
Cathodic Protection Service

Kansas City, Missouri
Industrial Coatings Engineering Co.

Long Beach, Calif.
Barnes & Delaney

Minneapolis, Minn.
Simcoe Equipment Co.

Philadelphia, Pa.
Harold N. Davis Co.

Plainfield, New Jersey
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St. Louis, Missouri
Shutt Process Equipment Co.

San Francisco, Calif.
Incandescent Supply Co.

Seattle, Washington
Farwest Corrosion Control Corp.

Seattle, Washington
Pacific Water Works Supply Co.

Northeast Region News

Water Conditioning Discussed by Panel

A panel discussion on "Miracle Water Conditioners" held at the January meeting of the Genesee Valley Section concluded that water conditioning, for any specific purpose, is an engineering problem requiring individual appraisal and solution and that the simple cure-all mechanical device for water conditioning has not been invented.

Panel members were G. L. Cox, Whitehead Metals Inc., R. N. Stenerson, Carrier Corp., D. K. Priest, Pfaudler-Permutit Co., R. C. Tucker, Niagara Mohawk Power Co., and D. Jordan, Hagan Chemicals & Controls, Inc.

Technical claims of several water conditioning devices were discussed, including the magnetic type that is claimed to eliminate scaling, soften water and prevent corrosion in numerous kinds of water systems. The panel stated that most manufacturers' literature has little technical data to support claims and is based on users' testimonials.

A panel member reported having seen one unit fail to produce the advertised claims, but the operator presumably thinks he is getting good service.

Another device creates the appearance of retarded scale build-up because more frequent blow-downs of the boiler system are specified, but corrosion had not been reduced as claimed, according to another panel member.

Consensus of the panel comments and discussion indicated that evidence based upon known and accepted principles shows that these devices cannot accomplish what is claimed of them. The panel suggested that prospective users should examine the situation thoroughly before making an investment or a replacement of a proven chemical means of water treatment.

This problem is discussed in the following literature: Eliassen, Skrinde and Davis, "Experimental Performance of Miracle Water Conditions," Journal American Water Works Association, Vol. 50, No. 10, Oct., 1958; Welder and Partridge, "Practical Performance of Water Conditioning Gadgets," Industrial & Engineering Chemistry, Vol. 46, No. 5, May, 1954.

•

Niagara Frontier Section held an afternoon meeting in March at the Buffalo Forge Company, Buffalo, N. Y. The meeting's topic was "Corrosion of Materials" and was given before the Buffalo Forge Student Engineering Group.

•

Kanawha Valley Section appointed a committee to stimulate interest in corrosion work among high school and college students by inviting selected students to section meetings. Committee members are W. H. Williams, chairman, Jack Bates and Lewis Wright.

Section officers for 1959 recently elected are Chairman W. G. Mathews of Columbian Carbon Co., Vice Chairman K. R. Gosnell of Union Carbide Chemicals Co., Secretary William J. Foster and Treasurer R. L. Davisson of Union Carbide Chemicals Co.

Art Erickson of Peoples Natural Gas Co., Pittsburgh, Pa., spoke on Coatings for Cathodic Protection of Underground Pipelines at the March meeting.

•

Central New York Section held a dinner-dance for its March meeting. The February meeting featured a talk by Charles Thomson of Budd Co., Philadelphia, Pa., on Radiographic Weld Inspection. Paul Noble of Republic Steel showed one of his company's movies on coatings.

New officers recently elected are Chairman Hendric Van Droeffleaar, Du Pont of Canada, Waitland, Ont., and Vice Chairman Gordon Watt of James Watt Co., Syracuse, N. Y.

•

Genesee Valley Section heard David Priest of Pfaudler-Permutit, Rochester, N. Y., present preliminary results of the salt corrosion tests in which the section cooperated. The tests were run to determine whether inhibitors placed in salt for city streets would decrease corrosion on automobiles. The cities involved were Buffalo and Syracuse where no inhibitor is used and Rochester, where inhibitors are used. Fifty panels measuring 6 x 3½ inches for mounting under automobiles and trucks were sent to each city. Preliminary results indicate that corrosion is decreased when an inhibitor is used in the street salts.

•

Lehigh Valley Section had a talk on precoating surface preparation of steel by Joseph Bigos of U. S. Steel at its March meeting.

A field trip through the research laboratories of Carpenter Steel Company is planned for the May 16 meeting.

Southeast Region News

Miami Section has set Nov. 16-20 as the dates for its 1959 Key Biscayne Corrosion Control Short Course to be held at the Key Biscayne Hotel in Miami. Joe Prime, Jr., has been appointed short course director and John Burns, secretary.

•

Carolinas Section officers for 1959 are Chairman Ray G. Penrose, Aluminum Company of America, Charlotte, N. C., Vice Chairman Robert H. Gardner, Jr., Koppers Co., Inc., Charlotte, N. C., and Secretary-Treasurer David B. Anderson, International Nickel Co., Inc., Wrightsville Beach, N. C.

The section's April 17 meeting will feature a talk by T. P. May on recent corrosion studies at the Kure Beach Harbor Island testing stations.

•

Over 226,000 copies of the NACE technical committee reports have been distributed.

•

Deadline for news stories in CORROSION is the tenth of the month.

•

CORROSION's index appears in December.

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Union-type end connectors, full-flow, quick opening, non-porous, never stick or gall, lightweight. ½" to 4" I.P.S.

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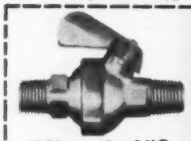

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Teflon sealed, non-sticking full-flow cock for corrosion-service applications. ⅛" and ¼" I.P.S. and hose connectors.

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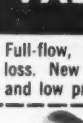

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North Central Region News

Chicago Section will send a subscription each year of CORROSION to the John Crerar Library in Chicago, starting with 1960. The action was in response to an appeal from the library for Chicago scientific societies to help the library with rising costs. The Crerar Library is regarded as a valuable source of technical information to engineers and scientists in the Chicago area, according to Section Chairman H. C. Boone.

K. R. Walston of Standard Oil of Indiana will speak at the April 21 section meeting on How to Paint Tanks Economically.

SECTION CALENDAR

April

2 West Kansas Section.
6 North Texas Section.
7 Shreveport Section. Caddo Hotel.
8 Metropolitan New York Section. Military Park Hotel, Newark, N. J. Discussion of various types of rectifiers.
8 San Francisco Bay Area Section. Spenger's Fish Grotto. Berkeley. Volatile Corrosion Inhibitors, by Aaron Wachter, Shell Development Corp.
14 Baltimore-Washington Section. Film and speaker on corrosion problems in nuclear reactors.
16 Detroit Section. Accelerated Testing of Protective Coatings, by Bill Cushing, Carboline Company, St. Louis.
17 Carolinas Section. Recent Corrosion Studies at the Kure Beach Harbor Island Testing Stations, by T. P. May.
21 Chicago Section. How to Paint Tanks Economically, by K. R. Walston, Standard Oil Co. (Ind.).
21 Cleveland Section. Cleveland Engineering and Scientific Center. Protective Coatings, by Stanley Lopata.
28 Panhandle Section.
30 Teche Section. Petroleum Club.

May

5 Shreveport Section. Caddo Hotel.
7 West Kansas Section.
13 Greater Boston Section. Water Treatment, by Rolf Eliassen, MIT.
16 Lehigh Valley Section. Field trip through research laboratories at Carpenter Steel Company.
19 Cleveland Section. Cleveland Engineering and Scientific Center. Panel discussion led by R. Weast.
19 Chicago Section. Corrosion Problems in Water Treatment, by John R. Baylis, Chicago City Water Dept.
21 Kanawha Valley Section. Parkersburg, W. Va. Solid Plastic Materials.
21 Teche Section. Petroleum Club.
21 Detroit Section. Metallography—A Tool in Corrosion Research, by W. K. Boyd, Battelle Research Institute.
25 Atlanta Section.
26 Panhandle Section.

Detroit Section will have the topic "Accelerated Testing of Protective Coatings" discussed by Bill Cushing, Carboline Co., St. Louis, at the April 16 section meeting.

W. K. Boyd of the Corrosion Research Division of Battelle Research Institute will discuss "Metallography—A Tool in Corrosion Research" at the May 21 meeting.

NATIONAL and REGIONAL MEETINGS and SHORT COURSES

1959

Sept. 29-30, Oct. 1—Western Region Conference. Bakersfield Inn, Bakersfield, Cal.

Oct. 1-2—Southeast Region. Jacksonville, Florida.

Oct. 5-7—Northeast Region. Lord Baltimore Hotel, Baltimore, Md.

Oct. 12-15—South Central Region Meeting, Denver, Col. Cosmopolitan Hotel.

Oct. 20-22—North Central Region, Cleveland.

1960

March 14-18—16th Annual Conference and 1960 Corrosion Show. Dallas, Texas, Memorial Auditorium.

Oct. 11-14—Northeast Region Meeting. Huntington, W. Va.

Oct. 19-20—North Central Region. Milwaukee.

Oct. 25-28—South Central Region Conference, Mayo Hotel, Tulsa.

1961

March—17th Annual Conference and 1961 Corrosion Show. Buffalo, N. Y., Hotel Statler.

Oct. 24-27—South Central Region Conference, Houston, Shamrock Hotel.

1962

March—18th Annual Conference and 1962 Corrosion Show. Kansas City, Municipal Auditorium.

October 16-19—South Central Region Conference, Hilton Hotel, San Antonio, Texas.

SHORT COURSES

1959

June 2-4—Teche Section. Corrosion Control Short Course, Southwestern Louisiana Institute, Lafayette.

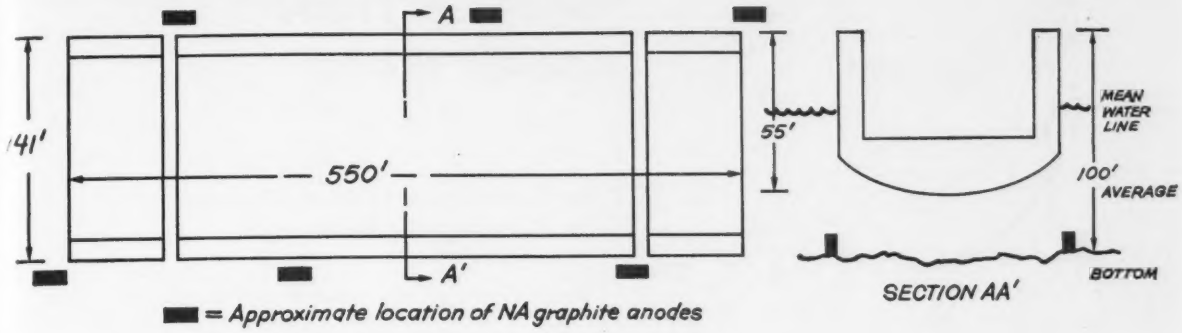
June 2-4—Appalachian Underground Corrosion Short Course, West Virginia University, Morgantown.

June 22-26—Massachusetts Institute of Technology Advanced Short Course in Fundamentals of Corrosion Reactions and Corrosion Control, Cambridge.

December 7-11—University of Illinois Corrosion Control Short Course. Urbana Campus.

Anode notes from

National Carbon Company



■ = Approximate location of NA graphite anodes

PROTECTION OF 90,000 SQUARE FEET OF STEEL IN SEA WATER

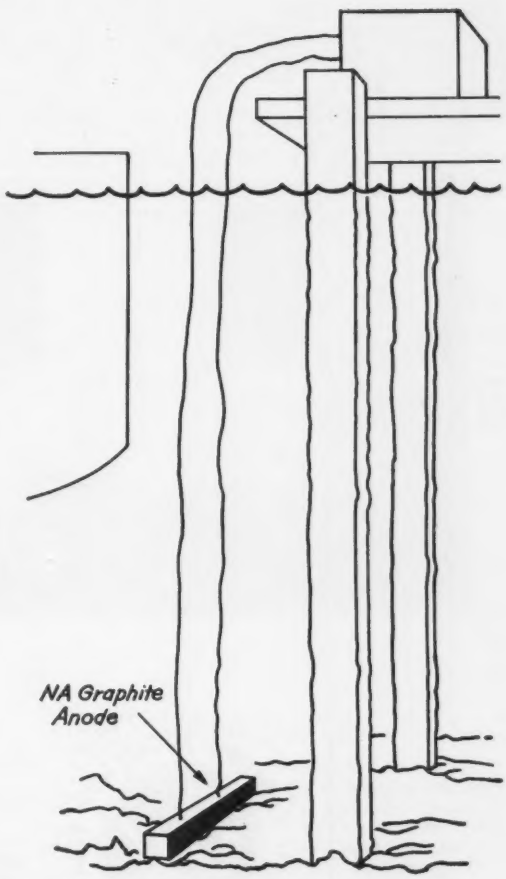
A corrosion engineer was given the problem of protecting a floating dry dock in a salt water harbor.

After a corrosion survey, the engineer decided to use impressed current cathodic protection and "National" NA graphite anodes for the submerged outside surface.

Six 12″ x 12″ x 72″ NA graphite anodes, located as shown above on the harbor bottom, are connected to the positive sides of six rectifiers. Each anode is discharging approximately 110 amps at 8.5 volts.

Large (approximately 600 lbs.) rectangular anodes were chosen to minimize movement on the bottom with tide currents. Two 125′ leads of 4/0 rubber insulated cable provide a low resistant path between anodes and rectifiers.

The system has been in operation since 1955. Semi-annual inspections show complete protection with negative potential readings to a copper-copper sulphate reference cell greater than 0.85 volts.



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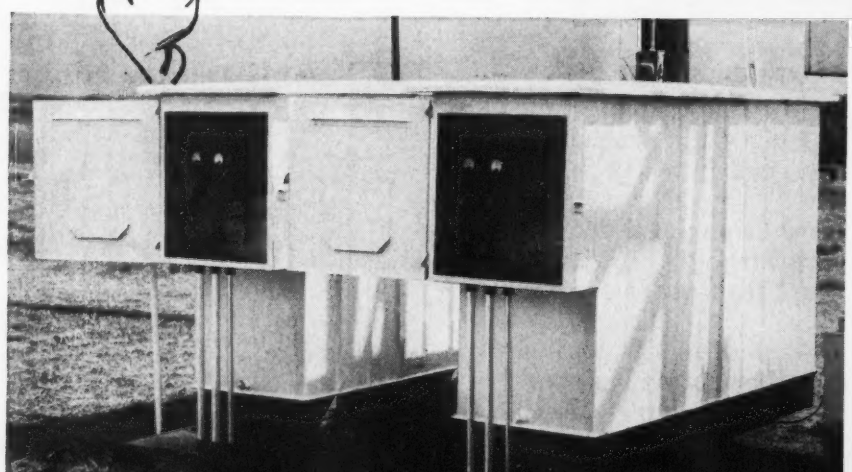

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GENERAL NEWS

Israel Is Establishing Marine Corrosion Station

A marine corrosion testing station is being established at Elath in Israel. It will cooperate with International Nickel's Harbor Island Test Station in North Carolina. Operations are scheduled to begin this summer.

General conditions at the Gulf of Elath are 15 percent higher salinity than the Mediterranean, average sea water temperature of 25 C and intense marine fouling.

Persons interested in testing products at the station should contact D. Spector, NACE member, at Citrus House, 22 Harakevet St., Tel Aviv, Israel.

Houston Paint Convention To Be Held April 23-25

Several papers of probable interest to corrosion engineers will be given during the 17th Annual Southwestern Paint Convention to be held April 23-25 at the Shamrock-Hilton Hotel, Houston.

The papers are "Use of Silicone Organics in Maintenance and Trim Enamels," "Liquid Epoxy Resins for Coatings," "Polyamides in Solventless Coating Systems," "Polyesters for Surface Coatings" and "Technique for Applying Thick Paint Films."

Corrosion Conference Set At New Hampshire College

Gordon Research Conferences on corrosion will be held July 20-24 at Colby Junior College, New London, N. H. A conference on physical metallurgy is scheduled for June 22-26.

Topics for the corrosion conference are Nature of Metal Seurfaces as Revealed by Friction and Wear Measurements, Kinetics of Oxidation Processes and Inhibition of Corrosion Reactions.

Additional information can be obtained by writing W. George Parks, Director, Department of Chemistry, University of Rhode Island, Kingston, R. I.

ASME Design Engineering Conference to Be May 25

Material selection in design, mechanical aspects of design, and power and control in design will be featured at the 4th annual Design Engineering Conference May 25-28 in Philadelphia. The conference is sponsored by the machine design division of ASME.

Some of the papers to be presented are "Engineering Design Overseas," "Latest Development in Plastics for High Temperature Service" and "Latest Developments in Metals and Ceramics for High Temperature Service."

SOIL BURIAL TEST PROGRAM conducted by Bell Telephone Laboratories has included the burial of about 11,000 pieces of telephone equipment in the damp soil near Bainbridge, Ga. The smaller samples are attached as shown above to a polyethylene plastic rod for burial at 18 and 6-inch depths. The metal strips at the upper end of the rod are identification tags which will be above ground. Purpose of the test is to determine how well the equipment withstands exposure to damp soil with no protection.

Telephone Equipment Pieces Are Buried For Long-Term Soil Deterioration Test

About 11,000 pieces of telephone equipment have been buried outside Bainbridge, Ga., by Bell Telephone Laboratories in a long-term soil test to determine the effect of dampness and high biological activity.

Batches of the equipment will be dug up at 1, 2, 4, 8, 16 and 32-year intervals. The equipment has been buried without any form of protection.

Buried samples included about 9000 small laminates, adhesives, plastics and various types of rubber, mounted on vertical polyethylene plastic rods in a six-acre tract. Almost 2000 insulated wire coils with voltage on half of them were buried two feet below ground surface. Larger equipment buried included several 200-foot lengths of cable connected to electrical power and laid in undulating lines to test reaction at different depths.

The test stems from the company's growing interest in buried telephone wires and cables without the extra protection provided by conduits. Recent damage to telephone cables by wind and ice storms has indicated the need for a more nearly storm-proof cable system.

Bell Telephone plans to conduct another test in the southwest where the same materials will be tested under the alkaline soil conditions to compare the results with the damp soil of Georgia.

Corrosion Short Course Held Recently in Belgium

A corrosion control short course sponsored by the Belgian Society of Industrial Chemistry was given February 9-12 in Brussels. Subjects included basic corrosion control theories, conditions for effective control of corrosion on steel by paints and industrial applications of corrosion control principles.

German Pipe Line

A 240-mile crude oil trunk pipe line has been completed between Wilhelmshaven and Wesseling, Germany. Oil velocity through the line will be 5 mph.

Galvanizers Committee

The steel industry's Galvanizers Committee sponsored by the American Zinc Institute will hold its 40th meeting April 23-24 at the Drake Hotel in Chicago.

Library Association Celebrates 50th Year

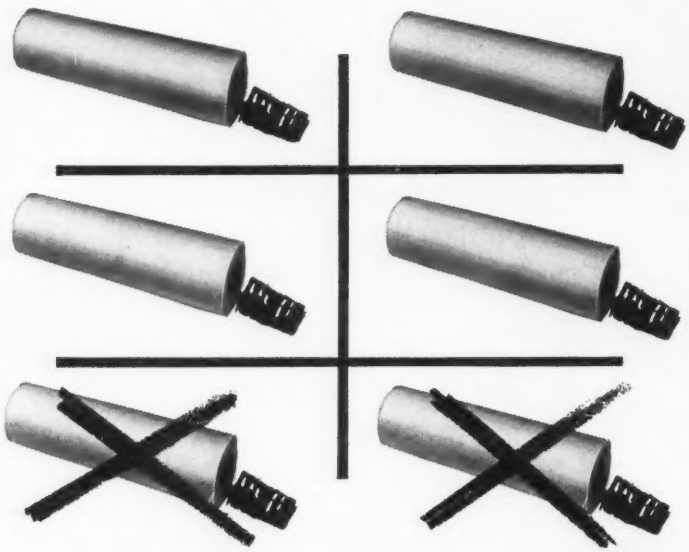
The Special Libraries Association celebrates its 50th anniversary in 1959. Some of the periodicals published by the association are the following:

Technical Book Review Index: Provides citations and quotations from book reviews, appearing in 800 scientific and technical publications. It is issued ten times a year and serves as a check list and index. Subscription in the United States is $7.50; elsewhere, $8.00.

Translation Monthly: Up-to-date listings of translations currently deposited in the SLA Translation Center. All languages are covered, including Russian. Cumulative indexes are published twice a year. It is published and distributed for SLA by the John Crerar Library, 86 East Randolph St., Chicago. Subscription is $5.00.

Refinery Air Pollution Report Now Available

Results of a 30-month survey of air pollution from petroleum refineries is available from the Los Angeles County Air Pollution Control District, 434 S. San Pedro St., Los Angeles 13, at $3.00 per copy. This is the ninth and final report prepared by the joint federal, state and district project established to evaluate petroleum refining emissions in the Los Angeles area.



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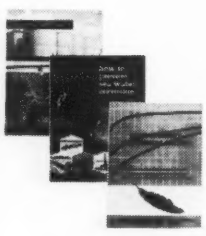

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BOOK NEWS

Technical Translations. Published semimonthly by the Office of Technical Services, U. S. Department of Commerce, Washington 25, D. C. Subscription rates: $12 a year ($4 additional for foreign mailing). Single copy price: 60 cents.

Provides a central source of information on translated technical literature available to science and industry. Lists abstract translations available from the publisher, Special Libraries Association Translation Center, cooperating foreign governments, educational institutions and private sources. Special Libraries Association's periodical, Translation Monthly, will be incorporated in this new publication.

Research Highlights of the National Bureau of Standards. Publication 226, 138 pages. Available from Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C. Per Copy, 45 cents.

The Bureau's 1958 annual report which gives the highlights of research and development programs at the Bureau. Includes a review of the Bureau's services to science and industry through testing and calibration of measuring instruments and through the distribution of standard samples.

Materials for Nuclear Reactors. Bernard Kopelman, editor. 424 pages, 6 x 9 inches, cloth. 1959. McGraw-Hill Book Company, Inc., 327 West 41st St., New York 36, N. Y. Per Copy, $12.

A systematic treatment of the atomic fuel cycle and of the preparation, properties and behavior of important materials in reactors. Contains 157 illustrations.

Thirty writers describe the use cycle of uranium from ore to the chemical reprocessing of irradiated, spent fuel elements.

Chapter 6, Fluid Materials, contains two sections of probable interest to corrosion engineers: "Corrosion Problems in Water" by C. C. Gregg and "Corrosion Problems in Liquids Other Than Water and Gases" by F. E. Bowman.

Atzheft—Verfahren zur Schliffherstellung und Gefugeentwicklung fur die Metallographie. By Angelica Schrader. (In German). 50 pages. 6x8¼ inches, cloth. 1957. Gebruder Borntraeger, Verlagsbuchhandlung, Berlin-Nikolassee/an der Rehwiese 4. Per Copy, DM 7,80.

Presents procedure for grinding preparation and joint development for the metal industry. Includes instructions for grinding preparation and classification of etching solutions, electrolytic polishing, electrolytic etching, list of chemicals and a bibliography.

PERIODICALS

Tetsu-to-Hagane Abstracts. (In English). Published annually by the Iron and Steel Institute of Japan, Naka-14-go-kan, Marunouchi-2-chome, Chiyodaku, Tokyo, Japan. Per copy $1.00.

Abstracts in English from the Journal of the Iron and Steel Institute of Japan covering iron and steel engineering and research in Japan. Issue No. 6, covering 1956, is now available.

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ASM May 4-6 Conference

Metallurgical Frontiers will be the theme of the ASM Southern Metals Conference to be held May 4-6 in Augusta, Ga. Topics to be discussed include coatings for corrosion protection, new frontiers in welding, modern metal removing techniques and concepts in solid feul power reactors.

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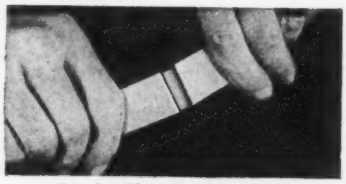

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JOBBER INQUIRIES INVITED

ASEE-ASTM Symposium to Discuss Teaching Problem

The problem of teaching materials to engineering students with already heavy college schedules will be discussed at a symposium of the ASTM Annual Meeting in Atlantic City, N. J., June 22-26. The symposium is sponsored jointly by ASTM and the American Society for Engineering Education.

Speakers at the symposium will present ASTM views and viewpoints of educators and industries. Frank L. LaQue, ASTM senior vice president and NACE member, will present the ASTM viewpoint.

The symposium will be concluded by a summary or survey of projected degrees in materials engineering now being considered or initiated in several engineering schools.

New Physical Science Curriculum Is Started

A new physical science curriculum has been started at Stevens Institute of Technology, Hoboken, N. J. Thirty-three students enrolled in the new curriculum will take basic courses in mathematics, chemistry, physics and some engineering sciences before taking advanced work in such areas as physical chemistry, atomic physics and differential equations.

These students also will study history, literature, philosophy under the Department of Humanities.

The curriculum leads to the Bachelor of Science, preparing the student for graduate work or careers in mathematics, chemistry or physics.

Translation Service

Translations of any article on iron and steel manufacture in foreign technical literature can be obtained from the British Iron and Steel Industry Translation Service, Iron and Steel Institute, 4 Grosvenor Gardens, London S.W.1. If more than one company or person requests a translation of the same article, the cost can be shared.

Research Management

Problems of managing research will be discussed at the Annual Industrial Research Conference to be held May 31-June 5. The conference is sponsored by the Department of Industrial Engineering at Columbia University and will be held at Columbia's Harriman Campus.

Waste Treatment Course

The fourth short course on theory and design of biological waste treatment will be held June 15-20 by Manhattan College Civil Engineering Department. Objective of the course is to present bio-oxidation fundamentals as a framework for analysis, design and operation of biological waste treatment facilities.

16mm Film on Plastic Uses

"The Architectural Uses of Plastics," a new 16mm sound film in color, is available on free loan basis from Swank Motion Pictures, Inc., 621 North Skinker Blvd., St. Louis 5, Mo.

The film is presented by Monsanto Chemical Company.

Electrochemical Society

The Electrochemical Society has scheduled its 115th national meeting at Philadelphia's Sheraton Hotel for May 3-7. Program details and registration information are given in the society's Journal, March issue.

Welding Conference

The 40th Annual Convention and Welding Show of the American Welding Society is being held April 6-10 in Chicago. Technical sessions are being held at the Sherman Hotel; the Welding Show at the International Amphitheatre.

Lubrication Society

The 14th Annual Meeting and Lubrication Exhibit of the American Society of Lubrication Engineers will be held in Buffalo, N. Y., on April 21-23 at the Hotel Statler. The Annual Short Course in Lubrication Engineering will be given also.

Lead Industries Meeting

Lead Industries Association will hold its 31st Annual Meeting April 22-23 at Chicago's Drake Hotel. A joint meeting with the American Zinc Institute will be held April 23.

•

South Central Region Meeting has been set for October 12-15 in Denver.

•

North Central Region Meeting will be held in Cleveland, October 20-22.

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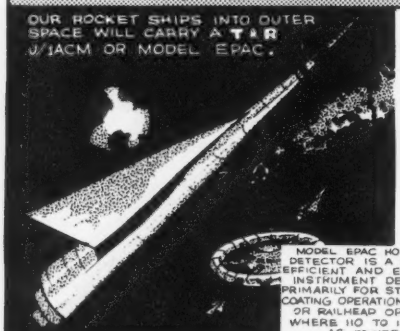

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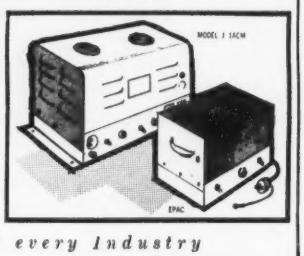

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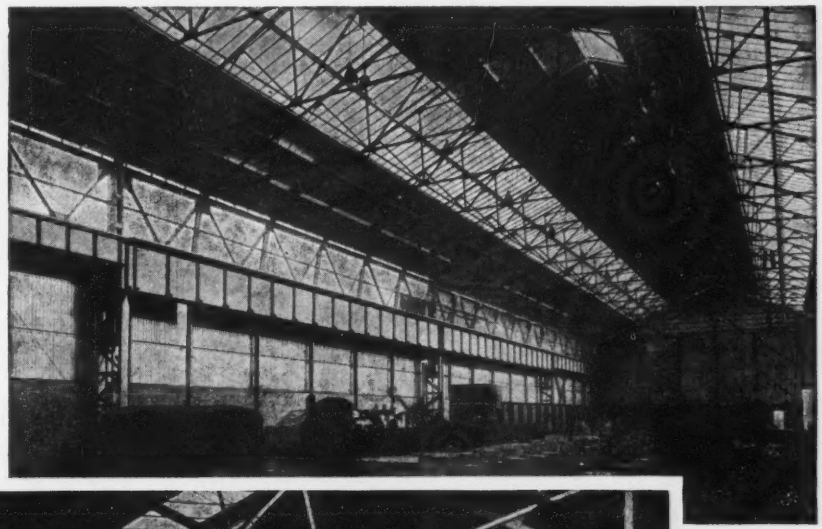
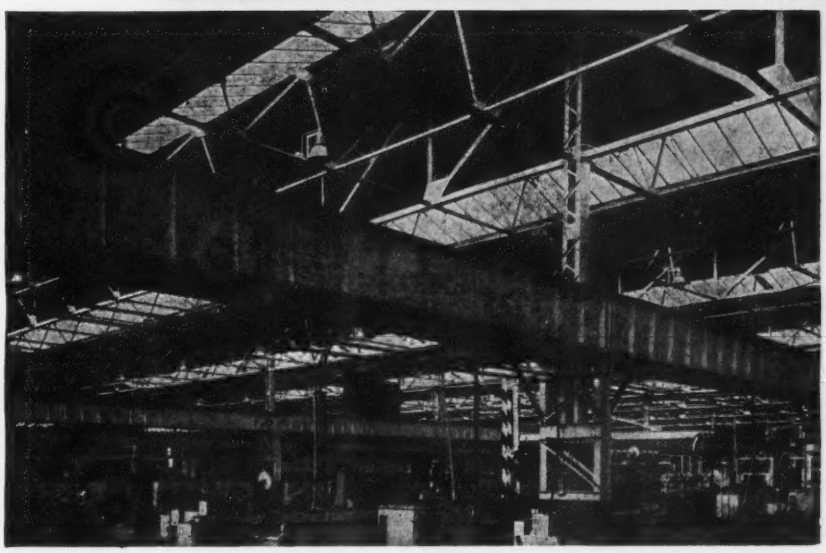


Steelwork in new mill painted with DRYGALV



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Equipment Services NEW PRODUCTS Materials Literature

Aluminum

Aluminum Laboratories Limited, Kingston, Ont., Canada, report that aluminum transmission towers were used in 1953-54 on the single circuit line over Kildala Pass. This was probably the first use of aluminum transmission towers.

•

High Purity Casting Alloy A356 manufactured by Kaiser Aluminum, 919 North Michigan Ave., Chicago 11, is described in a new brochure available from the company. Specifications, properties and performance data are given.

•

Heat Exchanger Fabrication with aluminum is summarized in a booklet available from Reynolds Metals Co., Dept. PRD-2, Box 2346, Richmond, Va. Designed to give information needed by heating engineers, fabricators and industrial purchasers, the booklet gives aluminum heat transfer rates, temperature properties, tensile strengths, tolerances, costs, corrosion resistance and charts on stress values, pressures, thermal expansion and conductivity and friction factors.

Bonding

Vinyl Plastic to Steel bonding with cement A-978-B made by B. F. Goodrich Industrial Products Company, Akron, Ohio, is reported to stretch 35 percent without weakening the bond or damaging the vinyl coating. Other tests on the cement bonding were in boiling water for 30 minutes, seven day exposure to 200 F and 1000 hours storage at 160 F.

•

Ball Bonding, a new technique for bonding aircraft components has been developed by Twin Coach Co., Buffalo, N. Y. Aluminum balls are used to eliminate voids by providing uniform application of pressure during the bonding process, consequently giving more uniform and rapid heat distribution.

•

Adhesive Bonding, sandwich construction and structural plastics are subjects covered in issues of Narmco Engineering Topics, a bimonthly publication available from Narmco Resins and Coatings Co., 600 Victoria St., Costa Mesa, Cal. It is distributed without charge to engineers, designers and technical personnel.

Cathodic Protection

Broyles Engineering Corporation has established a sales office and warehouse at Tulsa under M. E. Maddox, corrosion engineer. Engineering work on cathodic protection systems and materials out of stock will be available. The address is P. O. Box 4593.

Cleaning

Heat Exchanger Tube Bundles can be cleaned by a bundle-jetting tool developed by Dowell Corp., Tulsa, Okla. The cleaning tool rotates the bundle as water or chemical solvents are jetted onto the bundle, thus avoiding the erosion caused by sandblasting.

•

Striplite, a single dry salt, non-acid material for removing rust, scale and black oxide from steel is being manufactured by Du-Lite Chemical Corp., Middletown, Conn. A test bath is used to determine the solution strength needed for a particular stripping job.

•

New Tool Jets for chemical cleaning of storage tanks is being produced by Dowell Division of Dow Chemical Co., Tulsa, Okla. A modified version of Dowell's Zingger, it is designed for use in small tanks, horizontal, cylindrical tanks and tank cars. It uses high velocity streams of chemical solvents to remove scale, sludge and other deposits from internal tank surfaces.

Coatings—Glass

Porcelain Enamel on aluminum is reported to increase the metal's rigidity and to give resistance to impact, chemicals, abrasion, heat, fading, streaking and staining. This vitreous enamel coating is described in a brochure available from Argosy Industries, Inc., North Main & Grove St., Middletown, Conn.

Coatings—Organic

Coatings designed especially for petroleum refineries to prevent corrosion at sub-zero to elevated temperatures are described in a manual available from Xzit Chemical Co., Hoboken, N. J. Subjects covered include refractory protective coatings, chemical water treatments, solvent cleaning methods, soot, firescale and slag eradicators and chemical treatments for combustion and refining cycles.

•

Permiteco, Inc., is a new company formed when the assets of the Permite Paint Division of Aluminum Industries, Inc., were purchased. Permiteco is located at 1100 East Monument Ave., Dayton 2, Ohio.

•

Bakelite Epoxy Resin has successfully protected laboratory furniture from the attack of chemical fumes and spillage. The coatings were supplied by C. W. Haynes Laboratories, Inc., Springfield, Mass., for the lab furniture manufactured by Doane & Williams, Inc., 955 Chicopee St., Williamansett, Mass.

Expansion Joints

Stainless Steel Bellows Expansion Joints in 3 to 30-inch pipe sizes are being manufactured by Tube Turns Division of Chemetron Corp., Louisville, Ky. The joints are designed for applications to 275 psi and 750 F.

•

Garlock Packing Co., 433 Main St., Palmyra, N. Y., has issued a bulletin on rubber, Neoprene and Teflon-line expansion joints, their construction, sizes and uses.

Filters

Stainless Steel Electrofining unit has been installed by Petreco Division of Petrolite Corp., 369 Marshall Ave., St. Louis 19, Mo. The unit will remove slurry from an aluminum chloride catalyzed alkylation process. Preliminary pilot runs reduced volume percent from 0.7 to 0.03.

•

Air Washers made of rigid polyvinyl chloride made by Industrial Plastic Fabricators, Inc., Norwood, Mass, are available in capacities from 1000 to 54,000 cubic feet per minute. They are designed to handle acids, alkalies and corrosive fumes and are reported to clean 96 to 99 percent of contaminants that are instantaneously soluble in water, such as chromic acid, sulphuric acid, caustics, etc.

Inhibitors

Rodine 220, a new organic inhibitor for use with hydrochloric acid, is being produced by Amchem Products, Inc., Ambler, Pa., formerly American Chemical Paint Co. It is designed to protect steel and alloy steels exposed to muriatic acid at 200 F to 300 F.

Instruments

Drying Recorder, manufactured by Eastern Precision Tool & Gage Co., 451 Lehigh Ave, Union, N. J., is a new device to give precise measurement of drying time of paints, varnish, lacquer, enamel or other chemical products. It will also give data on film physical properties, adhesion through drying and tendency to wrinkle.

•

Electro-Hygrometer, a portable instrument developed by Labline, Inc., 3070 W. Grand Ave., Chicago 22, indicates relative humidity electronically from as far away as 100 feet. No tubes, conversion charts or thermometers are needed. Humidities from 30 to 95 percent within 32 F to 180 F temperatures can be indicated by the instrument.

•

Permeter, a perspiration meter to measure hand perspiration, is available from Ameresco Inc., 7 Center Ave., Little Falls, N. J. It is designed to check workers' perspiration in assembly departments where corrosion is a hazard.

Insulation

Prochind Insulating Joints, a prefabricated joint made by Prochind, n. 3 Via Lazzaretto, Milano, Italy, is inserted along pipe by welding, threaded connections or flanges. The joints are mechanically sealed for leak-proof service under high pressure and high tensile stress.

•

Glass Fiber Insulations are described in a product design brochure available from L-O-F Glass Fibers Co., 1810 Madison Ave., Toledo 1. Thermal and acoustical performance is also discussed.

(Continued on Page 92)

International Petroleum Exposition

5th

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NEW YORK, N. Y. May 30-June 5

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PERU: International Gas Lift Company, Apartado 71, Talara
TRINIDAD: Neal and Massy, Ltd., Port of Spain, P.O. Box 544

NEW PRODUCTS

(Continued From Page 90)

New Plants

Linde Company, division of Union Carbide Corp., will build a large liquid oxygen and nitrogen producing plant near Huntsville, Ala., in the vicinity of the Redstone Arsenal.

•

Vinyl Chloride Monomer production unit that will be fully automated is being installed at the Calvert City, Ky., plant of B. F. Goodrich Chemical Company. It will use a digital system to operate a chemical process specifically designed for computer control.

•

Sodium and Chlorine Production has begun at Du Pont's new Memphis, Tenn., plant. The company's capacity for these chemicals, used in high energy fuels, metal descaling and chemical processing, will be increased about 50 percent.

•

New Research and Development Plant is being built by Carborundum Co., Niagara Falls, N. Y. It will house facilities for development of new products that require ultra-high temperature processing techniques.

Non-Metallics

Globe Valves made of Karbate impervious graphite with a non-rotating spindle for more effective packing were among the equipment for corrosive service displayed at the 15th annual NACE Conference in Chicago last month, by National Carbon Co., 30 East 42nd St., New York City.

•

Reinforced Ceramics that resist gas erosion at extreme temperatures and eliminate structural failure caused by heat shock have been developed by Avco Research and Advanced Development Division at Wilmington, Mass. Called Avcoite, the new material can be fabricated for rocket nozzles, nozzle components and other applications subjected to very high temperatures.

Plastics

Molded Rubber Pails made with Du Pont Neoprene are being made by Cauchotex Industries, Inc., 44 Whitehall St., New York 4, N. Y. Named the Fortex NPL 80-18, the pail is designed to resist strong acids, gasoline, kerosene, jet fuels, oils, greases, brake fluid, paints, chemicals, etc. Its capacity is 18 quarts.

•

Heat Resistant Hose, made by the Thermoid Division, H. K. Porter Co., Inc., Tacony and Comly Streets, Philadelphia, Pa., is being extruded and fabricated in large diameter sizes from Du Pont's new heat and fluid resistant elastomer.

•

SRK Plastic Pipe, designed for new service line installations and insertion through existing corroded lines, is described in a catalog sheet (Adv. 1042) available from Republic Steel, Republic Building, Cleveland 1, Ohio.

•

New Affiliate of American Viscose Corp., and Sun Oil Co., named AviSun Corp., 1608 Walnut St., Philadelphia 3, Pa., will be a commercial supplier of polypropylene. To be in production by mid-summer, the new corporation will manufacture, process and sell resins, film, fibers, elastomers, surface coatings and adhesives using olefin polymers or copolymers with other materials.

•

Estane VC, a new thermoplastic elastomer developed by B. F. Goodrich Chemical Co., Akron, Ohio, is reported to have high tensile strength at high ultimate elongation, solvent resistancy to gasoline, low air permeability and abrasion resistance. It is available in the form of tough, rubbery granules which are converted into end products by milling, calendering, extrusion or molding.

•

Teflon Products made by Pennsylvania Fluorocarbon Co., Inc., 1115 N. 38th St., Philadelphia 4, Pa., are described in a brochure available from the company. Included are spaghetti tubing, flexible tubing, rods, instrument tubing, rubing connectors and extruded shapes.

•

Structural Fibers, Inc., Fifth Ave., Chardon, Ohio, offers seamless, reinforced plastic tanks in four standard sizes: 6 to 13-inch diameters and 32 to 54-inch heights. The tanks are designed to eliminate dents and ruptures under shock and to resist corrosion and contamination.

•

Repair Kit of liquid and plastic compounds to repair fractures, tears, holes and dents in metal, wood, plastic, concrete, ceramic and other materials is being produced by Taylor and Art, Inc., Plastics, 1710 E. 12th St., Oakland 6, Cal.

•

Serseal Chemical Blankets for use on acid pickling baths is available from Amchem Products, Inc., Ambler, Pa. Bath heating requirements have been reduced to 70 percent.

•

Frost Engineering Service Co., Huntington Park, Cal., has been appointed California distributor of Safe-T-Clad products manufactured by the Seamless Rubber Co., New Haven, Conn.

•

Teflon Sheets, rods, tubes, strips and slabs are being manufactured by Cadillac Plastic & Chemical Company at its new plant in Warren, Mich.

•

New Plastic Named PL-11 is a transparent acrylic-type thermoplastic polymer for injection molding and extrusion with high heat resistance developed by J. T. Baker Chemical Co., Phillipsburg, N. J. Similar in mechanical and optical properties to polymethyl methacrylate, it has a heat distortion temperature of about 250 F.

•

Plastic Piping System Technology is described in Handbook C-159 prepared by Chemtrol, 10872 Stanford Ave., Lynwood, Cal. Installation and coupling methods, plastic pipe standards, temperature and working pressure chart for Chemtrol valves and a table show effect of 280 chemicals on dive plastics are included.

Tantalum and Titanium

National Research Corp., 70 Memorial Drive, Cambridge 42, Mass., has formed a new Metals Division to consolidate production and marketing of NRC's high purity tantalum, tantalum alloys and other rare metals.

•

Brochure on Titanium intended as a reference for engineers, metallurgists, and designers is available from Harvey Aluminum, 19200 S. Western Ave., Torrance, Cal.

•

Sharp Bending of Titanium Tubing is now possible by modification of standard bending techniques developed by Dreisler Industrial Corp., Paterson, N. J. The work was done on grade A-40 soft-annealed, commercially pure titanium tubing produced by Superior Tube Co., Norristown, Pa. The tubing is bent on angles to 90 degrees with radii equal to that of the outside diameter in a variety of configurations.

Tubing

Stainless Steel Tubing for main surface condensers in power plants is covered in a technical report available from Alloy Tube Division of Carpenter Steel Co., Union, N. J.

•

Heat Resistant Tubes and coil forms that remain physically stable and without loss of insulation value at 1100 F are now made by a new technique of spiral winding inorganic bonded muscovite mica paper. Information is available from Resinite Corp., Dept CNH, 2035 W. Charleston St., Chicago 44.

Valves

Color Brochures describing the advantage of a new hydraulically controlled stainless steel ball cock is available from Fluidmaster, Inc., Anaheim, Cal. The brochures give the positive on-off control sequence and lists the advantages of corrosion resistance, automatic anti-sipon and instant shut-off.

•

Replaceable Discs and Seats made of Teflon for better corrosion resistance service are part of the features of the stainless steel valves described in Bulletin No. 11 available from Alloy Steel Products Co., Inc., Linden, N. J.

MEN in the NEWS

L. C. Ferris recently joined the staff of Cunat & Cunat, Ingenieurs Conseils, 6 Rue de Hanovre, Paris 2, France, a Franco-American firm of consulting architects and engineers. Formerly he was associated with the firm of F. A. Hughes & Co., Ltd.

•

W. Rex Bell, Jr., NACE member and president of Chemicals and Materials Corp., 3229 Wabash Ave., Terre Haute, Ind., was elected recently to the corporation's board of directors.

•

Archie C. Anderson, NACE member, has been named technical director of A. O. Smith Corporation's new reinforced plastics division, Milwaukee, Wis.

•

H. Maurice Banta has been appointed assistant director of research and technology for United States Steel's National Tube Division, Pittsburgh, Pa.

(Continued on Page 93)

MEN in the NEWS

(Continued From Page 92)

John M. Blocher, Jr., has been appointed chief of Battelle Memorial Institute's inorganic chemistry and chemical engineering division at Columbus, Ohio.

•

Peter E. Caron has been named manager of By-Product Sales Department of American Smelting and Refining Co., 120 Broadway, New York 5, N. Y.

•

N. R. Legge and **F. E. Condo** have been appointed heads of two new research departments (synthetic rubber and thermoplastics) created at Shell Development Company's Emeryville, Cal., Research Center.

•

Gilbert L. Cox, NACE member, has joined the staff of Whitehead Metals, Inc., 181 Winton Road North, Rochester, N. Y. He has been named technical manager.

•

Arthur R. Edwards has been named Chicago sales representative for the Plastics Products Division of B. F. Goodrich Industrial Products Co. He will have an office at the company's Chicago district office, 4646 West Lake Street.

•

Two NACE members, **William S. Ewing** and **Allen M. Smith II,** and Thomas A. Hanna have joined the Corrosion Engineering Products Sales Department of Pennsalt Chemicals Corp., 3 Penn Center, Philadelphia 2, Pa.

•

Karl L. Fetters, NACE corporate representative, was elected vice president of purchasing for Youngstown Sheet and Tube Co., Youngstown, Ohio.

•

John J. Gilman, metallurgist with General Electric Co., Schenectady, N. Y., received two awards at the AIME San Francisco conference recently: the Mathewson Gold Metal and the Rossiter W. Raymond Award, the latter was awarded to Mr. Gilman in 1956 also. The awards are given for outstanding technical papers contributing to metallurgical science.

•

Robert Gelin has been appointed assistant to the vice president at Structural Fibers, Inc., Chardon, Ohio.

•

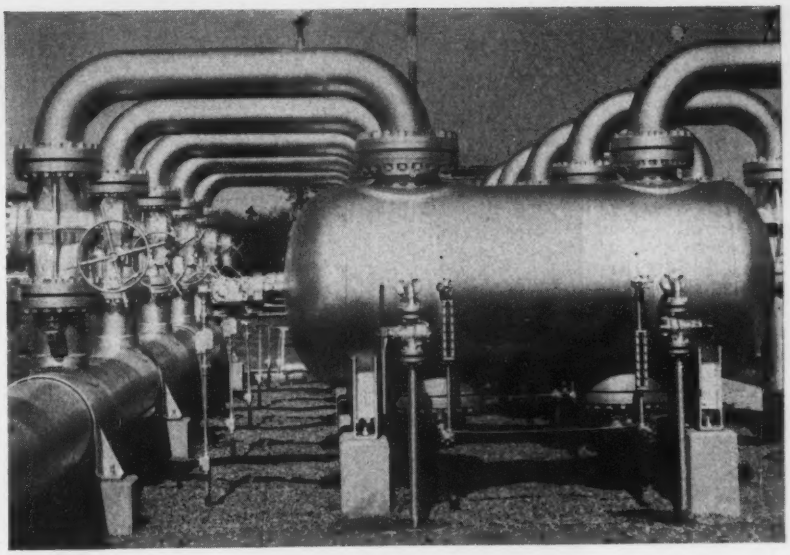
Dennis Chemical Co., 2701 Papin St., St. Louis 3, Mo., has elected **Sidney Dennis** president and **Frank Gollub** vice president of research and development. New appointments include **Milton Carlie** as sales manager and **Marvin Wool** as chief chemist.

•

Robert D. James has been assigned as field service engineer in A. M. Byers Company's Houston division office.

•

Edward A. Loria has been appointed as development manager of high temperature and corrosion resistant alloys for Climax Molybdenum Co., 500 Fifth Ave., New York 36, N. Y. He will coordinate developments involving molybdenum in steels and alloys for aircraft, missiles, nuclear power, chemical processing and other heat and corrosion resistant applications.



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With its many deep recesses, exposed edges, narrow crevices, inaccessible surfaces, and tricky shapes, this gas-transmission equipment presents a typical problem when it comes to combatting corrosion. Coating such objects as this by any method other than spray leaves edges thin, crevices inadequately coated, and many vital spots insufficiently protected.

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Four Carboline maintenance coating systems, thoroughly proven in service, are recommended for specific corrosive environments. Each system is "keyed" or tailor-made for a particular type of job. And each is designed to provide maximum protection for the longest time at the lowest cost per square foot per year of service.

CONDITION 1

Mild fumes, splash or spillage of most acids and alkalies. Continuous temperatures to 150°F. where maximum surface preparation is not practical. Structural steel, pipe, walls, equipment exteriors. **Carboline CS-200 (all vinyl) system.**

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Severe fumes, splash or spillage of acids and alkalies. Continuous temperatures to 180°F. Structural steel, equipment exteriors, concrete walls. **Carboline Epoxy 188.**

CONDITION 3

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CONDITION 4

Economical, easily applied maintenance system for mild chemical fumes. Requires minimum surface preparation. Continuous temperatures to 200°F. Can be used over old paints. **Carboline Epoxy 110.**

Write for newly released Chart No. 5 showing comparative ratings of coating systems by physical and chemical properties—an engineering aid in choosing the most efficient protection. Also ask for technical data and recommendations on Carboline maintenance systems.

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TECHNICAL TOPICS

Materials Selection and Design Problems In a Nickel-Cobalt Extraction Plant*

Abstract

Materials selection and design problems in connection with the design of the Cuban American Nickel Company plants at Moa Bay and New Orleans are discussed. These include handling ore slurries, the leaching system, pumps, process vessel linings, agitation systems and heat recovery units.

Materials involved include stainless steel, lead linings, acid brick, carbon brick, Teflon, titanium and ceramics. Design problems involved vessels handling slurries, slurry pumps, steam bleed lines, special flanging and valves. Because the process involves handling abrasive slurries leached with sulfuric acid at high temperatures, corrosion problems were extensive. 8.10.3

NICKEL AND cobalt ores are mined at the Moa Bay, Cuba deposits of Cuban American Nickel Company by open cut methods. Because the ore in place contains 30 to 35 percent moisture and the leaching operation does not necessitate drying the ore, the material is handled as a slurry throughout the system.

At the mine the ore is mixed with water to yield a free-flowing slurry of about 25 to 30 percent solids. After screening, the slurry runs by gravity to the plant area and flows into thickeners which bring the slurry to about 45 to 50 percent solids.

During the leaching process, the thickened slurry is preheated with recovered low-pressure steam to 160 to 180 F and pumped into reactors where high pressure steam is injected for final heating. Ninety-eight percent sulfuric acid is added as reagent. After leaching, the slurry is flashed to atmospheric pressure.

The pregnant leach liquor is recovered and barren tailings washed free from soluble values in a standard six-stage counter-current decantation system. The liquor is neutralized by treatment with coral slurry derived from a nearby offshore reef. Finally the concentrate is prepared by precipitating the nickel and cobalt, with minor impurities, from the neutralized leach liquor with hydrogen sulfide, using slightly elevated temperatures and moderate pressures. Sulfides precipitated are granular and settle rapidly, are washed free of barren liquor in small thickeners and then are ready for transport to the refinery.

Problems Involved

The heart of the process and the focal point of this article is the leaching system. In Figure 1 a schematic diagram of the system is given, showing the principal features. Many serious problems were involved in converting the basic chemical information (that the nickel and cobalt could be extracted with sulfuric acid at high temperatures) to actual operation on a commerical scale.

First, the possible operating temperature range dictated that pressures could not be less than 400 psi. Leaching rates were greatly improved by using temperatures where pressures were above 500 psi. This means that the heavy, viscous slurry must be pumped at discharge pressures above 500 psi. Then the slurry must be heated to operating temperature.

The high pressure pumps are diaphragm pumps of the pressure-exchange type. That is, the primary pumping is done on a suitable hydraulic fluid which activates the diaphragm to pump the slurry. The design finally selected was developed principally by Humble Oil and Refining Co. These pumps are pressure-fed by rubber lined centrifugals. The slurry is heated by direct absorption of steam.

Once the ore is heated to leaching temperature, the reagent acid must be added and permitted to react; then the reacted material must be discharged from the system. Agitation of the reactor stages is provided by high pressure steam injection. Heat is recovered by indirect heat exchange to generate 30 psi steam used elsewhere in the process.

Agitation with high-pressure steam involves the use of a draft-tube (See Figure 2) and its advantages depend on further use of the high pressure steam bled off the reactors to heat the feed slurry. This system has several important advantages over mechanical agigators:

It avoids the limitation on vessel design that shafting problems in large mechanical agitators would impose.

It avoids the heavy power consumption and maintenance costs of mechanical agitators.

First cost is much less.

Corrosive System

The corrosive system is as follows: An aqueous suspension of fine iron ore (about 36 percent solids by weight), with the solution phase containing (in grams/liter) Ni, 5 to 7; Co, 0.5 to 1.0; Fe, 1 to 5; Al, 3 to 8; Mg, 2 to 4; Mn, 3 to 4; Cr, 0.1 to 0.7 (in the form of chromate). Acid addition is equivalent to about 10 percent on the basis of the solution. However, reaction is rapid and residual acid seldom exceeds 3 percent. Temperature is 400 to 500 F, and agitation is moderate.

Extensive coupon tests, using every material available, were made. Only titanium and its commercial alloys showed completely satisfactory characteristics. Lead does not, strictly speaking, corrode in the system, but at 450 to 500 F—the region of operation—it is hardly strong enough to support its own weight. Nickel alloys, from the stainless steels to the Hastelloys, cannot survive in the system.

Titanium, while tolerable from a cost standpoint in places where light gauge material is acceptable such as draft-

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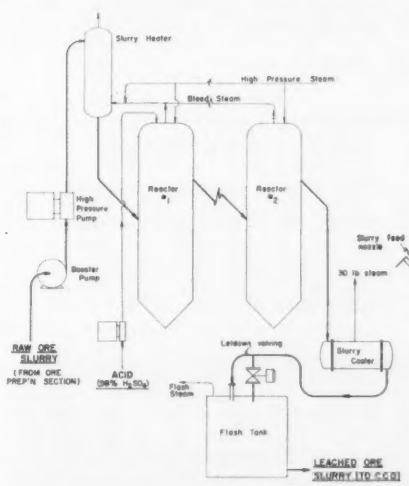


Figure 1—Ore leaching system.

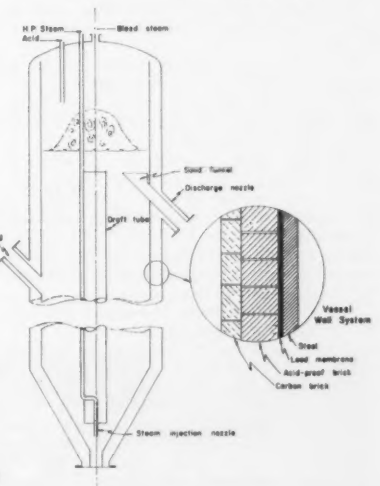


Figure 2—Main features of a leaching reactor.

★ Adapted from a paper titled "Nickel Extraction by the Moa Bay Process: A Problem in Materials Selection" by C. S. Simons, Cuban American Nickel Co., New Orleans, presented at a meeting of South Central Region, National Association of Corrosion Engineers, New Orleans, October 20-24, 1958. Published in condensed Form as "Novel Designs Tame Tough Corrosives," p. 130, Chemical Engineer, Jan. 26, 1959.

Materials Selection—

(Continued From Page 95)

tubes and pipings, could not be considered for the vessels.

Solution to the vessel lining problem is indicated in the inset to Figure 2. This system of lead-lined steel, acid-proof brick and carbon brick appears to be a fairly simple and obvious solution to the problem. Lead is commonly used in sulfuric acid systems and was found to be essentially corrosion-proof. This solution, however, represents a great deal of testing and observation. The summary report on this phase of the pilot plant work runs to over 150 pages.

Use of Lead in Construction

Some general observations of interest on this construction are:

1. Bare lead linings—homogeneously bonded to the shell—were desirable from an economic standpoint. Brick linings represent a substantial part of the vessel cost. Bare lead was rejected because it is vulnerable to mechanical damage and because, when subjected to cyclic heating and cooling it tends to develop cracks. This effect was noted on bear lead in the reaction vessels (subjected to full operating temperature) but was not apparent in the lead behind brick linings where temperatures were lower.

2. It is difficult to get a satisfactory lead bonding for service temperatures of 400 to 500 F. After a few heating-cooling cycles, unbonded areas show up as blisters. Particular care is needed in bonding over heavy sections, such as flanges where, because of the high total heat capacity, it is difficult to heat the base metal to bonding temperatures.

Lead Bonding Specifications

The study of lead-bonding resulted in a system regarded as essential for service under the severe conditions encountered. A brief summary of the specifications developed follows:

(a) Bonding **must** be by hand.

(b) A **clean** steel surface is essential. This means grinding with a carborundum wheel and immediate tinning before the ground surface has the opportunity to oxidize. Sand-blasting was avoided because of the possibility of sand grain inclusions. Acid washing must **not** be used.

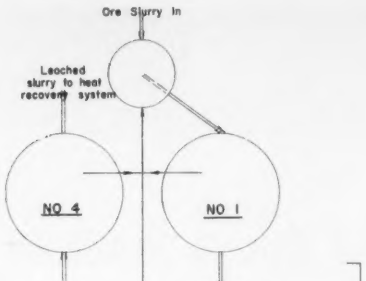
(c) Any good tin-lead solder may be used for tinning the 50-50 mix being preferred.

(d) Steel must be sufficiently hot to be thoroughly wet by the solder.

(e) Excess solder must be wiped off; this may mean two separate wipes with clean rags to remove all the excess.

(f) The lead should be chemical grade. While it has not been found definitely that small amounts of common contaminants (Sn, Bi, Sb, Ag, Cu, etc.) have any influence in bonding or subsequent service characteristics, lead meeting ASTM specification B-29 for Chemical lead is satisfactory.

All acid-proof brick is subject to some spalling in the system, a common condition where brick is used at high pressures. While carbon brick does not spall, it has no appreciable thermal insulating properties. Consequently acid brick was selected for internal thermal insulation and carbon brick was used to protect the acid brick. When a system of this type is used, outside steel surface temperature was 225 F when internal temperature was 475 F. Because the lead temperature is only a few degrees above the steel surface temperature, it is evident that the lead is not subject to excessively severe temperature cycling.



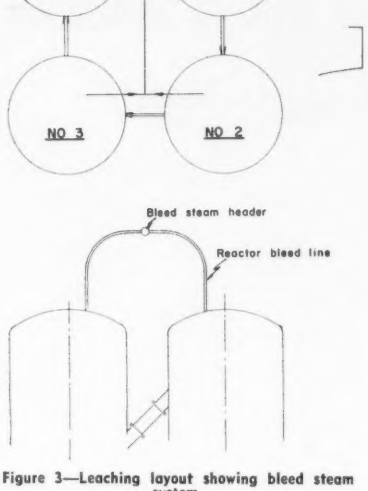


Figure 3—Leaching layout showing bleed steam system.

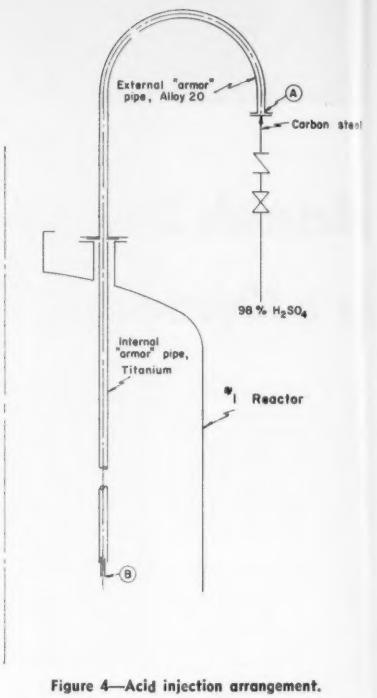


Figure 4—Acid injection arrangement.

None of the silicate mortars could be used because the system is not acid enough to produce a good cure. This seems to be anomalous, because the liquor under operating conditions is fiercely corrosive to virtually all alloys normally used for corrosion resistance. The steam phase ruled out silicates in the upper section entirely.

Because the system is oxidizing, synthetics of the furane type are attacked. While furane resins based on furfuryl alcohol were better than those based on furfuryl ketones neither were completely satisfactory. Epoxy resins are excellent, but very awkward to apply. The final cement system selected was furfuryl alcohol cement for the acid brick course; and for the face course, National Carbon C-6 cement, which is self-carbonizing when heated.

Bleed Steam System

Two unexpected problems arose in connection with the reaction vessel:

Figure 3 shows a schematic diagram of the bleed steam system. This is the steam injected into the vessels for agitation, which then must be drawn off for use in preheating the feed slurry. While the pilot unit was too small to attempt the recovery operation, an agitation-bleed system was used in the first continuous reactor built in Texas in 1954. The bleed piping was made of Type 316 stainless steel with standard fittings. The only difficulty with this bleed steam system was an erosion failure on the downstream side of an elbow located immediately above the reactor proper. The remainder of the piping appeared to be in excellent condition. This experience led to selection of Type 316 for the bleed steam system of the Braithwaite pilot plant where sharp changes in direction were avoided by using long radius bends.

After a short time in operation at Braithwaite, the bleed steam lines off the reactors unexpectedly developed a number of tiny leaks. Metallographers immediately identified the cause as stress corrosion cracking due to chlorides. This seemed incredible because there were only 30 to 50 ppm chlorides in the water used for slurrying the ore. The ore itself, by the very nature of its formation, could not contain more than trace amounts of chloride. Bleed steam sampled over a considerable period always contained 10 to 16 ppm chloride (expressed as NaCl); temperature was 475 F and failures occurred in **all** highly stressed zones (field-made bends which were not subsequently stress-relieved and in forged welding fittings used in the header) in both Type 316 and 316 ELC. Later tests showed Type 310 and Alloy 20 and 20 Cb unsatisfactory.

The result of this was specification of titanium for the bleed system of the commercial installation, dictated in part by the stress corrosion problem and because leached slurry **can** get into the bleed system. Because of stress corrosion, Type 316 could not be used; a much more expensive alloy was required. Therefore, there appeared to be ample justification to use titanium for full protection.

Figure 4 shows how the problem of getting the acid in continuously without interruptions due to equipment was solved. In the small pilot unit (Texas), glass pipe was used. An adaptor was made to go in the vessel nozzle to support the injection pipe and to seal it to the transfer line. The acid line and junction flange were water cooled to

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prevent corrosion of the transfer line due to heating from the reactor. This installation used pipe with a ¼-inch bore with a short nipple of ⅛-inch bore attached to the discharge end on the theory that higher exit velocities would improve mixing of acid and slurry. Whether this exit nipple had any beneficial effect in the expected way is not known, but it prevented the line from draining during periods of no flow.

Glass proved entirely too fragile at Braithwaite even with armor pipe to eliminate lateral stresses. Teflon tubing with a different adaptor but attached near the vessel nozzle flange failed because it is occasionally necessary to shut off acid flow momentarily. When this happened, the acid drained from the line and steam entered the tube. This diluted the acid remaining on the surface and heated the surface to reactor temperatures. The result was rapid dissolution of the adaptor and the metal line adjacent to it.

Figure 4 illustrates the solution worked out by the pilot plant staff which is the subject of a patent application. It employs a continuous Teflon tube from point A to point B, the terminus in the vessel. Externally to the vessel flange, a hairpin shape of Schedule 40 Alloy 20 pipe is used with enough clearance between the Teflon liner and the pipe to equalize pressure, but not enough to permit appreciable refluxing. Thus the Teflon is subjected only to the pressure of the fluid head loss within it. If acid flow is shut off, the line may drain up to the high point, and steam can reflux in this section. But this does not affect the inert Teflon and the connection at A cannot get hot nor can the diluted acid reach it.

Figure 5 shows the method of going through the vessel nozzle with the acid injection line. The Alloy 20 external pipe ends in a 3/16-inch plate flange welded to the pipe. The titanium armor pipe internal to the reactor is identical to this at its upper end. These are flanged together using a carbon steel blind, machined to fit, as a back-up flange. Seal is obtained with a spiral-wound gasket.

This figure also shows the method employed to protect the lead lining in the small vessel nozzles. Nozzle design for these vessels required close attention because of the severe service. The nozzle flange is machined out and an Alloy 20 overlay applied. The Alloy 20 forms the bearing surface for the seal gasket. The overlay is re-machined to the OD of the seal gasket and upset so that the lead lining can be brought over the flange. Finally a titanium thimble, with a top flange as support, is slipped in. This serves to protect the lead from mechanical damage and supports it against creep.

Heat Recovery Problems

Operation economy depends on recovery of a substantial part of the heat content of the leached slurry. The procedure selected was generation of low-pressure steam by indirect transfer. This was investigated in detail in the Braithwaite pilot plant to establish effective heat transfer coefficients and to determine whether fouling of the inside tube surface would be a problem. The prin-

(Continued on Page 98)

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cipal point of interest here was the information this investigation gave on velocity limitations.

The cooler-boiler, as the unit is called, is a shell-an-tube heat exchanger operated like a low-pressure fire-tube boiler. The tubes **have** to be titanium. It was found that the hot end (in the pilot plant unit, four tube passes with external return bends were used) experienced considerable erosion of the titanium at velocities of 8 to 10 ft/sec. This was not observed in the cold end. This effect led to the selection of 6 ft/sec as the maximum velocity permissable in the tubing and 4 ft/sec as the design velocity.

While there are several theories as to the cause of this difference in erosiveness in the same piping at the same **mass** rate, only the writer's personal opinion is given here. There is both a contraction in volume and an increase in viscosity between the hot end (at 400 to 450 F) and the cold end (at 250 to 275 F). This means the Reynolds number of the flow decreases by a considerable amount, perhaps a factor of ½ to ⅓, in going from the hot end to the cold. Because the Reynolds number is the criterion of turbulence, this is the important difference. This seems a more likely explanation than that the titanium itself is appreciably softer or less abrasion resistant at the higher of such a relatively narrow range of temperature.

The final point covered in this article is the problem of getting the leached material out of the system and back to atmospheric pressure.

The material is about 30 to 35 percent solids slurry of very fine iron oxide (predominantly) at 500 to 550 psi and it is, even after heat recovery, still above the atmospheric boiling point. This means it will flash. And a flashing, acid slurry taking a 500 lb pressure drop is a **very** difficult problem.

Ceramics Used in Chokes

The final solution, shown in part in Figure 6, represents the end of a long development program. Early in the work it was determined that conventional valving had no chance of success. Both Calera Mining Company at Garfield, Utah, and National Lead Company at Fredericktown, Missouri, have similar problems. While the final solution differs from theirs, they were helpful in pointing out lines of approach that undoubtedly shortened the period of development required to achieve a workable design.

A complete list of possible valve trim was explored and showed that the only materials with any life at all were fused alumina ceramics and Carborundum's Refrax. Nothing will withstand the abrasion of these slurries at high velocities for an appreciable time because their action is similar to grinding with jeweler's rouge suspended in acid. This meant that there was no real chance of throttling the flow to an exact rate, so development was aimed at finding a system wherein the valves could be operated off-on. In this way, the wear on the trim is confined to the instants of opening and closing, and the life of the trim becomes a function of the number of cycles of operation.

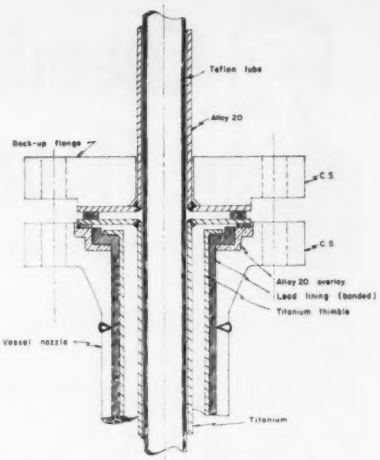

Figure 5—Detail diagram of vessel entry for acid injection.

Because ceramic is one of the most satisfactory trim materials available, it was decided that control should be built around standard oil field chokes, or Refrax chokes made to the same dimensions. Problems of valve body erosion, actual seat and plug construction and others were solved gradually.

Figure 6 is a diagram of the method of installing the chokes which control the flow. The scheme is to provide two chokes in parallel; one, sized to handle about 90 percent of normal total flow, forms the terminus of the discharge pipe from the slurry cooler-boilers; the other, sized to handle about 15 percent of normal total flow, is mounted below an off-on control valve actuated by surge level. The controlled flow by-pass line branches off the main line 15 inches above the pad flanges on the flash tanks. The main line is flanged upstream of the by-pass. This makes a compact assembly which can be removed easily by breaking out three flanges—the two pads and the main line—after which another unit is installed. The old unit may be removed for inspection and replacement of chokes.

Note that the choke section consists of two 6-inch choke sections cemented in a section of pipe. A necessary part of the assembly is the ceramic disc with a bore somewhat larger than the choke bore to prevent erosion of the support plate due to entry turbulence.

When metal temperature was kept below 250 F, a number of alloys gave satisfactory service in the leached slurry. Type 316 was used at Braithwaite, but completely satisfactory service in this case required waterjacketing the line. Thus the alloy selected for choke holders and control valve body is Alloy 20; this was found by testing to be twice as resistant to the system as Type 316. Although the chokes are cemented in the holders, back-up plates are used to give added assurance of choke retention. These support barrels may be used again to assemble replacement units.

This article covers superficially the materials selection problem of one section of one plant. There were other sections with other problems. It can be said quite fairly that the need for titanium provided by the commercialization of the Moa Bay process gave impetus to further development of that material for chemical process use. Needs of the Moa Bay plant provided much of the spark that induced the primary producers—such as Titanium Metals, Rem-Cru and Republic—and fabricators—such as Crane, Oremet, Nooter and Struthers-Wells—to develop techniques necessary for chemical plant equipment. Particularly noteworthy in this respect is Crane's work on valve design and Oremet's development of techniques for making the body castings for the 5-inch titanium block valves.

45 Technical 16mm Films Are Available From AEC

The AEC is making available for loan and purchase the 45 technical films used at the 1958 Geneva Conference. The 16mm films, most in color, cover major nuclear applications and research activities. A list of the films and offices where the films can be purchased or borrowed is available from the AEC office in Washington 25, D. C.

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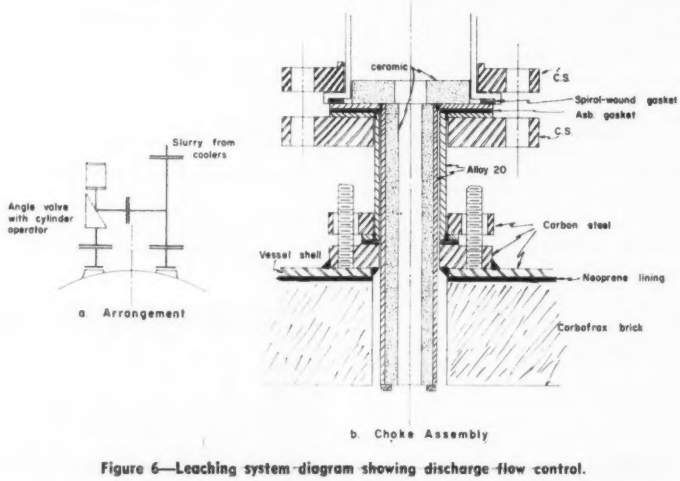
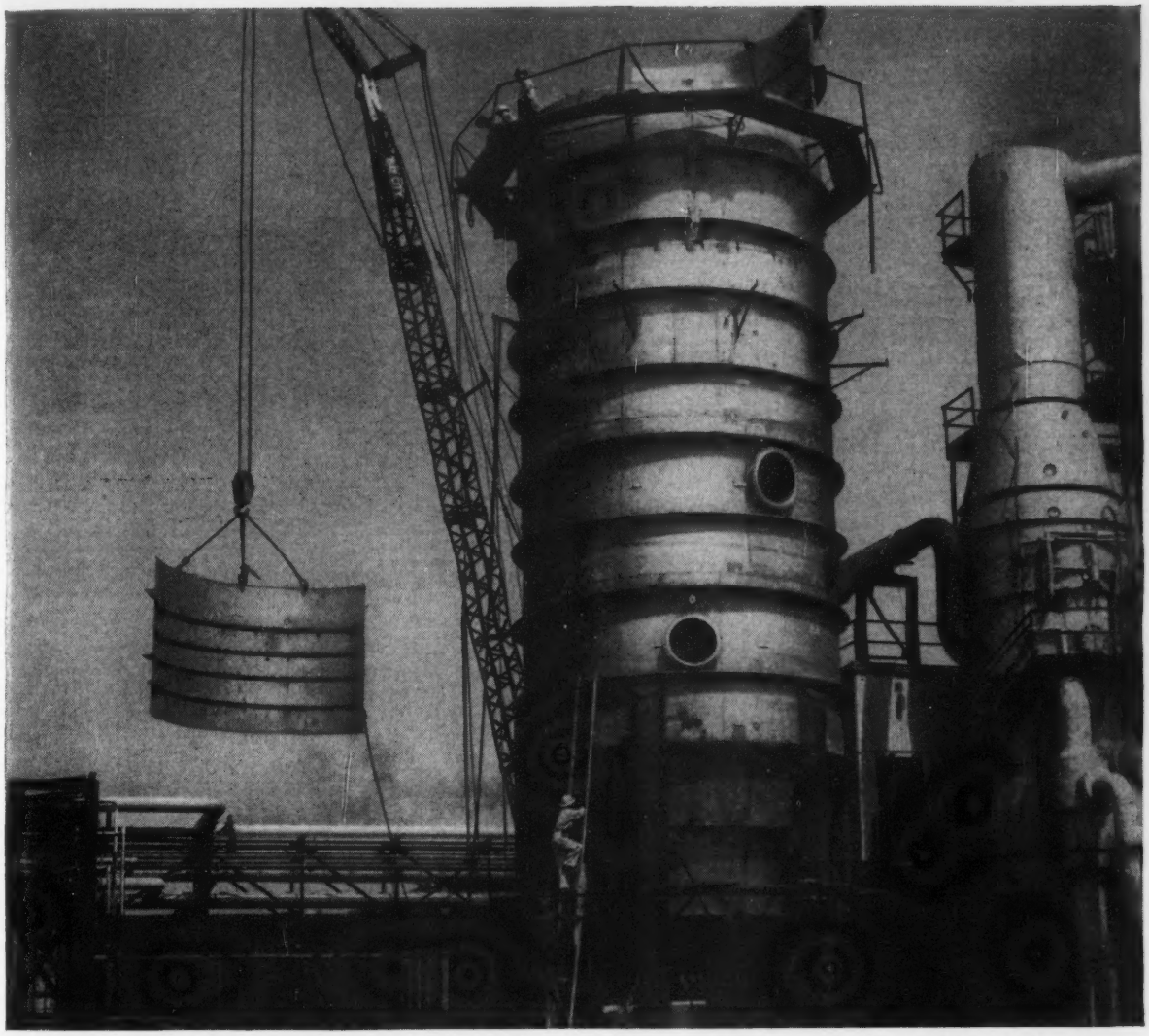


Figure 6—Leaching system diagram showing discharge flow control.



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New Formulations Give Versatility To Urethane Coatings*

Abstract

New formulations in the manufacture of polyurethanes have given wider application of these materials. Variations have been made in flexibility and hardness over a wide range of temperatures. Production of more stable isocyanates has eliminated the two package system and reduced the water reactivity that limited the use of older two package system. Other new formulations have been developed to improve chemical and solvent resistance, hardness and flexibility.

Case histories are given of several coating applications using the new formulations. 5.4.5

THE ELASTOMERIC nature of certain of the urethanes presents an interesting future for urethane rubber. The solid urethane elastomers have shown unusually high abrasion resistance, high tear strength and good shock absorption coupled with hardness, solvent resistance and good resistance to oxygen aging.[1] These rubbers are typical of other urethanes in that a wide range of flexibility and elasticity is possible by varying formulation components. These materials have been used successfully as gears, check valve seals and oil field parts such as valve inserts in high pressure pumps where they give four times the service life of other materials.

The largest market for these urethane rubbers is found overseas where their production is greater and engineering experience in the urethane field more extensive. However, the American market for urethane rubber is increasing. Urethanes are giving remarkable results in the fields of adhesives and textiles.

The polyurethanes as developed in Germany were unusual coating materials, exhibiting high orders of adhesion, gloss, water and solvent resistance, good electrical properties, low gas permeability and high chemical resistance.

Because of this versatility, the coating chemist found that great variations could be made in flexibility and hardness over a wide range of temperature conditions, as indicated in the following case histories:

Case One: A urethane coating on a water-gas generator showed no failure or metal corrosion after six years although it was subjected to corrosive gases and physical strain as a result of changing temperatures varying between freezing and 250 F.[2]

Case Two: The holds of several oil tankers have been protected with urethane coatings. These tankers carry alternately sour crudes and salt water ballast. The urethanes are withstanding these conditions.

Case Three: In a rubber accelerator plant, a urethane coating has successfully withstood the action of acid, amine and hydrogen sulfide vapors for over eight years. The corrosion engineer at this plant said other paints lasted less than two years in this atmosphere.

These three cases concern air dry, two component systems which have been improved.

Water and Solvent Sensitivity

The air dry urethane coating system has one disadvantage: sensitivity to water and solvents containing active hydrogen. An active hydrogen is defined in this instance as any hydrogen atom which may be replaced from a structure by metallic sodium. In the manufacture of foamed urethanes, this water sensitivity is an advantage and an important factor; this reaction generates the gas to create foam. In a coating this is undesirable. The foaming action with water creates pinholes and holidays in the film, or the entire mass of the coating in the container can react before application, causing the coating to become jelled or semi-jelled and unsuitable for application.

Because of this high reactivity with water, special care must be taken by the formulator and applicator to keep the materials as dry as possible. Even relative humidity becomes a critical factor in many cases. The formulator is further hindered in the selection of solvents for the system because extreme care must be taken to see that no solvents containing active hydrogens are present in the formulation.

Recent developments have produced a more stable isocyanate that not only overcomes the water reactivity objection but also makes possible the elimination of the two package system. Two package systems, after mixing, usually have a limited application life and require great care in measuring and mixing to produce the intended coating system. Overcured, undercured and spottily cured films result from too little or too much of the reactive component or from undermixing. Exact ratios of reactants can be blended during manufacture to insure maximum qualities of the materials in one package systems. However, this new coating system requires high temperature baking to produce a cured coating because of its high stability at room temperatures. Good adhesion is gained on steel, aluminum and other materials by the same preparation techniques used in other baking coatings. As is the case with baked phenolics and epoxy-phenolics, only operators having the necessary speecialized equipment can apply it.

New Formulations

From additional study of possible materials for reaction, new formulations have been developed which greatly improve chemical resistance, solvent resistance, hardness and flexibility. A baked urethane formulation superior to the German air dry is available today. This material has high flexibility and impact resistance found neither in baked phenolic nor epoxy-phenolic but having caustic, acid and solvent resistance equal to them in many cases. This baked urethane shows high permeation resistance in temperatures to 200 F and unusual resistance to underfilm corrosion creepage.

This baked urethane coating was designed originally for use on thin wall aluminum tubing in salt water disposal systems and flow lines protecting this tubing from salt water and other chemicals extremely corrosive to this metal. The flexibility of polyurethanes would make protection possible where phenolics and epoxy-phenolics had failed. The chemical resistant properties would afford protection where the flexible but less resistant coatings such as epoxy polyamides and oleoresinous compounds had failed. This material was approved for service by a major aluminum company after years of search for a suitable material. Subsequent field testing has shown this coating to have a definite use with tubular steel products also.

Tube Coatings

Six cases illustrate the effectiveness of polyurethane as a tube coating:

Case One: A coated pipe was given an 80-degree U-bend on a 5-foot radius with no damage to coating adhesion or to the coating itself. Thus, the coating permits the bending of pipe in the field for use in above-ground flow lines.

Case Two: In a pumping well, rods and tubing were coated. After one year, no sign of wear was found where rods had rubbed constanty against the tubing. Although the uncoated well pump was pugged with paraffin, only a thin film of paraffin was present on the polurethane coated surfaces. This paraffin had such little adhesion that it dropped off during handling. The operator thought that, if the rods had not been coated, paraffin would have prohibited removal of rods from the hole.

Case Three: A well in West Texas required weekly rod pulling jobs because of salt water corrosion. Coatings had been considered a poor risk in the past because of the lack of flexibility and impact resistance of existing coatings. A pay-out for a coating job was calculated at six months. Since the rods were coated with urethane, the well has been in operation 16 months.

Case Four: The urethane coating in a salt water disposal well is doing a satisfactory job after one year, according to the corrosion engineer in charge. Several hundred thousand feet of urethan coated tubing are in service without a coating failure recorded to date.

Case Five: Down-hole tubing in another well was coated inside and outside for corrosion prevention utilizing a total of 3400 feet of 1-inch tubing and 3400 feet of 2-inch tubing. After friction wrench make-up during running, no mechanical damage was present on the coated surfaces.

Case Six: This urethane has made possible the fabrication of down-hole water pumps of coated ferrous materials replacing bronze alloys at a cost saving of 50 percent.

The urethanes are a new material with versatility and unusual properties. The full potential of this material cannot be realized in the short space of a few years. Laboratory research is being conducted by many groups into further specialized uses for polyurethanes.

References

1. Richard C. Burck. Urethane Coatings. *Corrosion*, 13, 130 (1957) Dec.
2. Barnard A. Dombrow. Polyurethanes.

★ Extracted from a paper entitled "Polyurethanes—Plastics With An Oil Field Promise" by Barnard Goodman, Tube-Kote, Incorporated, Houston, presented at a meeting of the South Central Region, National Association of Corrosion Engineers, New Orleans, October 20-24, 1958.

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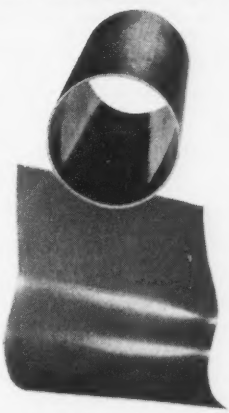

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Characteristics and Cost Factors

GLASS FIBER Reinforcement for COATINGS

Figure 1—Parallel reinforcement of textile strands in porous type glass fiber pipe wrap used for internal reinforcement of coatings that are machine applied and which require high tensile strengths during application.

GLASS FIBERS are being used to reinforce coal tar and asphalt coatings applied to underground steel pipes used for the transmission of water, crude oil, natural gas and other petroleum products.

One type of reinforcing fiber glass used in pipe wrappings is porous with parallel reinforcement of textile strands as shown in Figure 1. This type is used for internal reinforcement of coatings that are machine applied, particularly in over-the-ditch operations where high tensile strengths are required during application.

Resistances of Glass Fiber

The ability of fiber glass to resist impact is shown in Figure 2. The two pipes were stuck together. The top pipe was unreinforced and its coating spalled, leaving the metal exposed to corrosion. The reinforced coating on the bottom pipe was only bruised at the point of contact indicated by the arrow.

Another characteristic of glass fiber reinforcement is resistance to the atmosphere, shown in Figure 3. The two pipes were machine coated with the same coal tar enamel and exposed to the atmosphere for two years. The pipe on the left with the badly cracked coating had no reinforcement while the other had glass pipe wrap and showed little effect from the exposure.

Glass fiber is an effective reinforcing material in an underground corrosion protection system because it is resistant to the electrochemical attack of water, acids and alkali found in soils. Table 1 gives the softening point and resistance of pipe wrap glass fiber to water, acid and alkali in comparison to window glass and Pyrex laboratory glass.

Abstract

Uses of glass fibers as reinforcement in coal tar enamels for corrosion control of underground pipe is discussed. Resistance of glass fiber to impact, atmospheric conditions and chemical attack is given with data presented in tables to show the properties and cost factors of basic fiber types, mats and cloths. Possible future applications of other fiber types are discussed briefly also.

6.6.6

Glass fiber reinforcement must be produced at a cost low enough so that an adequate amount can be economically used on a given job. For example, the most commonly used fiber has a diameter of 0.0006-inch. There is approximately one pound of this glass fiber in each hundred square feet of underground pipe wrap. This amount of wrap sells for about 75 cents.

Another economical fiber type used for corrosion protection is the continuous textile strand. The most common example of this type is called the 150 1/0. This textile strand is made up of a bundle of parallel glass monofilaments, each being approximately 0.00038 inches in diameter. A bundle has 204 monofilaments and a diameter of 0.0069-inch. The continuous textile strand has higher tensile and impact strengths than the coarser monofilament.

Underground pipeline installation is estimated to cost in dollars per mile at 2500 times the pipe diameter in inches. A 10-inch pipe costs $25,000 per mile. The cost of glass pipe wrap for this diameter pipe is about $170 per mile, or less than one percent of the total cost.

Other Types of Glass Fibers

Several forms and combinations of glass fibers have been used successfully. One is the porous mat made up of monofilaments of 0.0006-inch diameter. A variation of this porous mat is one containing random reinforcing of textile strands to increase tensile strength in all directions. Another type has parallel reinforcing strands as shown in Figure 1. Another porous wrapper is made entirely of textile strands in a woven fabric, 20 yarns to the inch. After weaving, the fabric is treated to prevent weave distortion during the wrapping operation. This wrapper is used with either hot or cold applied coatings and usually is hand applied.

Machine and hand applications of glass fiber wrap are shown in Figures 4 and 5. Over-the-ditch application by machine of a saturated wrapper consisting of the monofilament mat filled with bitumen is shown in Figure 4. Figure 5 shows a typical hand application of the saturated textile cloth. The saturant is being softened with a torch to obtain good adhesion.

Glass Fiber Properties

Properties of monofilament porous mat and porous textile woven fabric are compared in Table 2. The textile fabric has less fiber length per dollar and lower fiber surface area per dollar than the porous mat. The fabric also has higher tensile strengths and higher impact resistance than the monofilament mat. Individual monofilaments have greater length and surface area, but when combined as textile strands, most of the surface area is not available as frictional resistance to the flow of the coating.

(Continued on Page 104)

★Extracted from a paper titled "Glass Fiber Reinforcing of Coatings" by B. A. Graham, Johns-Manville Fiber Glass, Inc., Toledo, Ohio, presented at a meeting of the North Central Region, National Association of Corrosion Engineers, Cincinnati, October 15-17, 1958.

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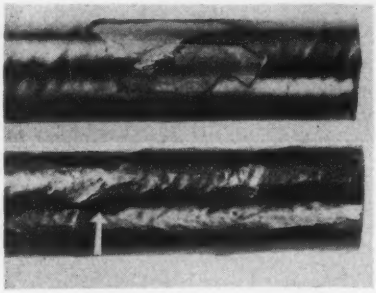

Figure 2—Impact resistance of a coating that has been reinforced with glass fibers. Two pipes shown were struck together so each would receive the same impact. Both pipes were coated with 3/32-inch of modified coal tar enamel. The unreinforced coating on the top pipe spalled, leaving the metal exposed. The reinforced coating of the bottom pipe was only bruised at the point of impact, indicated by the arrow.

TABLE 1—Comparison of Glass Types as Fibers

Property	Window Glass	Pipe Wrap Glass	Pyrex Glass
Softening Pt.°F.	1200	1280	1508
Loss 1 hr. Boiling Water	11.1%	2.0%	0.8%
Loss 1 hr. H_2SO_4 1 N....	6.2%	4.3%	0.8%
Loss 1 hr. NaOH 0.1 N..	20.0%	6.4%	40.0%

TABLE 2—Comparison of Mat and Cloth

Property	Pipe Wrap .020 in. Mat.	Woven-Treated Cloth 20 x 20
Glass Fiber, lb/square.....	.85	1.1
$/square.....	.85	3.00
Tensile lb/in.....	16	75.0
Fiber length/dollar/miles...	600	3
Fiber surface area/ dollar/sq. ft.....	500	29

Glass Fiber—

(Continued From Page 102)

Woven cloth is more expensive than the glass fiber. Cloth is woven by a textile manufacturer after the glass fiber is made by another manufacturer, then a third manufacturer usually treats the fabric. Monofilament pipe wrap mats are sold by the glass fiber manufacturers as a finished product.

Other Types of Fibers

Another basic fiber type is the B fiber staple used in many insulation applications such as under automobile hoods. The properties of this staple are compared to textile 150 1/0 and mat monofilament in Table 3. The unusual tensile strength and impact resistance of the textile strand may be used in future applications of non-woven fabrics for corrosion control.

One non-woven fabric being developed is made up entirely of resin bonded textile strands about three inches long. This is a thinner mat of the type used in plastic reinforcement. It is thin enough and porous enough for use as an internal reinforcement wrap in coal tar enamels.

Summary

Basic properties necessary for reinforcing found in glass fibers are chemical durability, high strength, high fiber length and high fiber surface area at low cost.

Only two forms of glass fibers have been used as reinforcement in coatings: The 0.0006-inch diameter monofilament mat and the woven textile fabrics, but many other basic glass fibers are available and other forms of assembling them are possible for future uses.

TABLE 3—Comparison of Basic Fiber Types

Property	B Staple	Mat Monofilament	Textile 150 1/0
Fiber diameter inches..	.00015	.00060	.00690
Fiber length miles/lb..	9800	600	8
Surface area sq ft/lb...	2000	500	79

Figure 3—Resistance of glass fiber reinforced coatings to atmospheric exposure. Both pipes were machine coated with the same coal tar enamel and then exposed to the atmosphere for two years. Pipe on the left had no coating reinforcement; the other had glass pipe wrap.

Technical Topics Scheduled for May

Comparative Effectiveness of Coatings for Offshore Drilling Structures, by C. G. Munger

High Temperature, High Pressure Testing of Organic Coatings for Oil and Gas Well Tubing, by Cordell Garner

Solving Some Oil Ash Corrosion Problems, by R. S. Norris

Figure 4—Saturated wrapper consisting of monofilament mat filled with bitumen is being machine applied in an over-the-ditch operation.

Figure 5—Typical hand application of a saturated textile cloth containing glass fibers. The saturant is being softened with a torch to obtain good adhesion.

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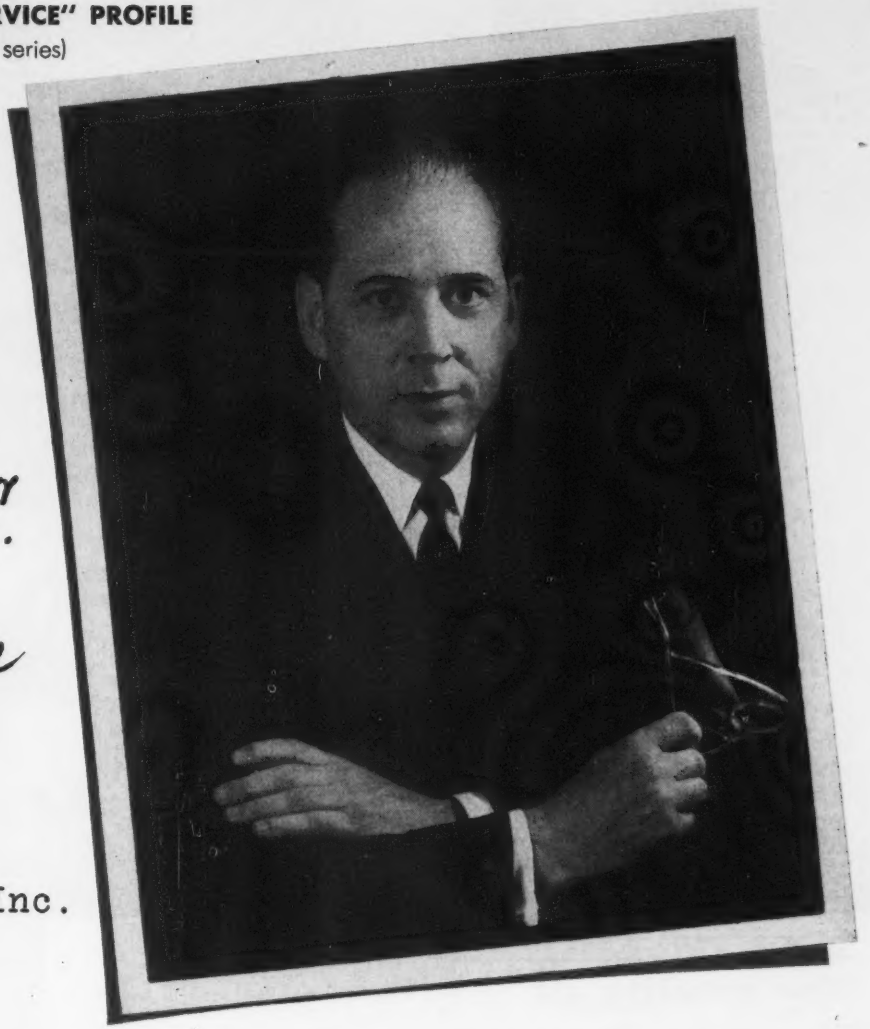

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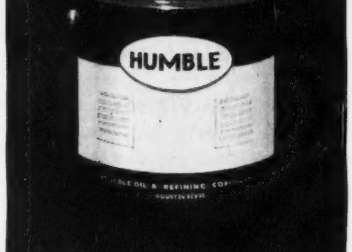

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CORROSION ABSTRACTS

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1. GENERAL

1.6 Books

1.6

Corrosion Symposium, Cologne, 15 May 1957. Metalloberfläche, 11, No. 11, 349-355 (1957).

Detailed resumes are given of the following papers presented at the meeting: Th. Heumann, Current Knowledge of Passivation and Surface-Film Formation; H. J. Engell, Studies on the Corrosion of Metals Forming Imperfect Surface-Films; C. Carius, The Significance of Oxygen in the Passivation and Corrosion of Chromium and Corrosion-Resistant Chromium and Chromium-Nickel Steels in Boiling Sulphuric Acid; L. Piatti, Passivation and Surface-Film Formation in Softened Water; D. Altenpohl, Formation and Properties of Surface Films Formed by the Interaction of Aluminum and Water; M. Weiner, Current Knowledge of the Corrosion of Condenser Tubes; H. Klas, Studies on Surface-Film Formation Resulting from Cathodic Polarization; M. Gräfen, The Influence of Surface-Film Formation on Stress Corrosion Cracking; A. Kutzelnigg, Corrosion Protection by Air Conditioning; H. Determann, Corrosion Protection During Sea Transport in Tropical Climates; O. Marsch, Corrosion of Metal Surfaces in Natural and Artificially Produced Tropical Climates.—MA. 15875

1.6, 6.3.18

The Metallurgy of Vanadium. W. Rostoker. Book, 1958, 185 pp. John Wiley and Sons, Inc.

Subjects covered include extractive metallurgy of vanadium, properties, corrosion and embrittlement, and vanadium as an alloy addition.—INCO. 15516

1.6, 4.7

Lithium Symposium—Analytical Procedures and High-Temperature Corrosion. Reading List E. E. Hoffman and D. H. Jansen. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., CF-57-10-6 (Rev.), January 20, 1958, 32 pp. Available from Office of Technical Services, Washington, D. C.

A list of 222 references pertaining to corrosion by molten lithium and related subjects was compiled in connection with a Lithium Symposium held at the Oak Ridge National Laboratory on August 7-8, 1957. This list is by no means exhaustive but it is thought to contain most of the information presently available on these subjects. In many cases the individual references will contain additional references. (auth) —NSA. 15452

1.7 Organized Studies of Corrosion

1.7.1

Scientific Achievements of TsNIITMASh in the Field of Materials and Technology of Mechanical Engineering. (In Russian.) I. R. Kryanin. Metalloved. i Obrabotka Metallov., No. 11, 56-65 (1957).

Kryanin reviews the work of the Russian Central Scientific Research Institute of Technology and Mechanical Engineering in developing new materials for machine construction, methods of testing materials for research or quality control (creep, fatigue, static, corrosion, erosion and non-destructive testing), production methods (vacuum-casting, working, welding and heat-treatment) and methods of improving service life.—MA. 15924

1.7.1, 3.8.2

The Fourth Conference on Electrochemistry. (In Russian.) B. N. Kabanov. J. Phys. Chem., USSR (Zhur. Fiz. Khim.), **31,** No. 2, 524-527 (1957).

The conference was held in Moscow in October 1956. One session was devoted to the influence of the double-layer formation on the kinetics of electrode processes, four sessions to hydrogen overvoltage and electrochemical reduction, three to electrochemical oxidation and passivation, four to electrodeposition and one session to electrochemical processes in non-ferrous metallurgy. Problems connected with the action of storage batteries were also discussed.—MA. 15886

1.7.1, 6.2.2, 6.2.3

A Half Century of Work in Corrosion of Iron and Steel. Committee A-5 Celebrates Fiftieth Anniversary. H. F. Hormann. Am. Soc. for Testing Materials Bull., No. 228, 17-20 (1958) Feb.

Recounts highlights in history of Am. Soc. for Testing Materials Committee A-5 on Corrosion of Iron and Steel, covering early work to determine difference in corrosion rates of iron and steel, effects of possible segregation in ingot, and influence of carbon and manganese contents of steel; numerous atmospheric corrosion tests of plates and wires to determine effectiveness of pigments as protective coatings, weight of zinc coatings, and influence of copper content in steel; immersion tests in city, brackish, mine and sea waters; comparison of relative corrosion rates with respect to atmospheric test location; investigation of copper-steel plates and silicon-steel plates fastened with rivets of different composition exposed to sea water; and work on causes of embrittlement of hot-galvanized steel. Present tests on atmospheric corrosion of carbon steel, alloy steels and nodular irons at Kure Beach and Newark, N. J., are mentioned.—INCO. 15523

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Corrosion Metallurgist-Chemical Engineer: B. S. Chemistry, 1949, chemical engineering, 9 years technical experience, 6 years as lead engineer for corrosion section. Technical papers, NACE: Symposium chairman, Nuclear Field. NACE Committees-vice chairman. Married, Age 32. CORROSION, 59—17

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Northeast Region Meeting will be held October 5-7, 1959, at Baltimore, Maryland.

•

South Central Region Meeting has been set for October 12-15 in Denver.

•

The Western Region Conference is scheduled for September 29-30, October 1, 1959, at Bakersfield, Cal.

•

North Central Region Meeting will be held in Cleveland, October 20-22.

2. TESTING

2.1 General

2.1.1

The Use of Statistics in Corrosion Research. G. M. W. MANN. *J. Applied Chem.*, **7**, No. 7, 419-424 (1957) July.

Principles underlying use of statistical calculation of probability and correlation and variance of results are outlined and their application to examination of corrosion problems is discussed. —INCO. 14704

2.1.1, 7.5.5, 4.4.6

A Statistical Evaluation of Corrosion in the Bottom of a Hydrocarbon Tank by Sampling. C. MEYER. *Rev. Inst. Francaise Petr.*, **12**, 834-863 (1957) July- August.

A statistical evaluation of thickness measurements made on cylindrical samples obtained by controlled sampling from the bottom of a 16.5 m dia., rather uniformly corroded hydrocarbon storage tank during service shows that in 20 years the bottom had become 0.70 mm thinner and that an extra thickness of 1.0 mm would be desirable for the bottom of a new tank. The results suggest that the relative frequency curves for non-corrosion and surface corrosion are symmetrical although flater than Gaussian distributions, while variations in pitting corrosion follow the Galton distribution law. 15371

2.2 On Location Tests

2.2.5, 1.7.1, 2.2.2

Long-Term Corrosion Tests on Painted Steel Exposed to Atmosphere. (In French.) Paul Nylen. Peintures, Pigments, Vernis, **34**, 116-117 (1958) March.

Corrosion Committee of Royal Academy of Sciences of Sweden has, since 1938, carried out several series of extended tests on plates painted with different types of paints and exposed to atmospheres in various parts of Sweden. Results are reported in terms of influence of climate and nature of paint on intrusion of rust.—MR. 16098

2.2.2, 5.4.5

Corrosion-Research Laboratories. Pt. XII. Ministry of Supply Outdoor-Exposure Station, Glencoed, Wales. Testing Paints by Outside Exposure. (Pts. I, II). C. R. Edwards and E. Jenkins. *Corrosion Technology*, **4**, Nos. 6, 7; 192-194, 245-250 (1957).

Pt. I. The site and equipment is described. Paint breakdown is measured by a single assessment.

Pt. II. Each specimen is assessed at fixed intervals. A scale of 5 degrees of failure is discussed and illustrated.—MA. 14933

2.3 Laboratory Methods and Tests

2.3.2

The Acetic Acid Salt Spray Test. J. H. Hooper. Bull. Inst. Metal Finishing, **8**, No. 1, 79-90 (1958) Spring.

Describes a large-scale investigation which was conducted to compare the relative merits of the 1% Acetic Acid

5% Salt Spray Test with other accelerated methods for evaluating the corrosion resistance of anodized aluminum and chromium-nickel-copper systems on steel, zinc and copper basis materials. A considerable number of specimens were tested by the three accelerated test methods and by outside exposure, including a wide range of anodic film thickness on high purity aluminum and super purity aluminum (99.99%) together with a wide thickness range of chromium-nickel-copper deposits upon steel, brass, and zinc basis materials. It is concluded that the 24 hour sulfur dioxide test is the most suitable for rapid assessment of chromium-nickel and chromium-nickel-copper deposits on steel, but that the hot or cold acetic tests were the most useful for evaluation of similar coatings on zinc base alloy or brass. In the case of anodized high purity and super purity aluminum, it is shown that a definite relationship exists between anodic film thickness and rate of breakdown, using the hot acetic acid method, but that the other accelerated methods are not very satisfactory. The work confirms the opinion of previous investigators, that the straight 5% salt spray is not suitable as an accelerated test for chromium-nickel-copper systems on any of the basis metals tested or for anodized aluminum, in that virtually no correlation exists between the rate and type of breakdown found when using the test and that which occurs under atmospheric exposure. (auth).—ALL. 15642

2.3.2

Corrosion Testing Methods. W. Wiederholt. Paper before Congres Européen de la Corrosion, Paris, November 1956. Corrosion et Anticorrosion, **6,** 80-100 (1958) March; Werkstoffe u. Korrosion, **9,** 133-146 (1958) March.

Detailed description of controllable, accelerated corrosion tests under laboratory conditions. Tests include humidity, sulfur dioxide, carbon dioxide, salt spray and immersion. Supporting data on steel, zinc, aluminum and copper plus nickel plus chromium plated steel are given.—INCO. 15600

2.3.2

Salt Spray Testing of Tinplated Copper. I. Corrosion Measurement by Chemical Porosity Test. Martin S. Frant. Plating, **45,** 157-160 (1958) Feb.

Correlation of visual results with the porosity test, and examples of its use in determining corrosion rates, comparing various types of salt-spray, determining the effect of plating thickness on corrosion resistance, comparing different plating solutions, and the effect of reflowing (melting) the tin coating. —BTR. 15458

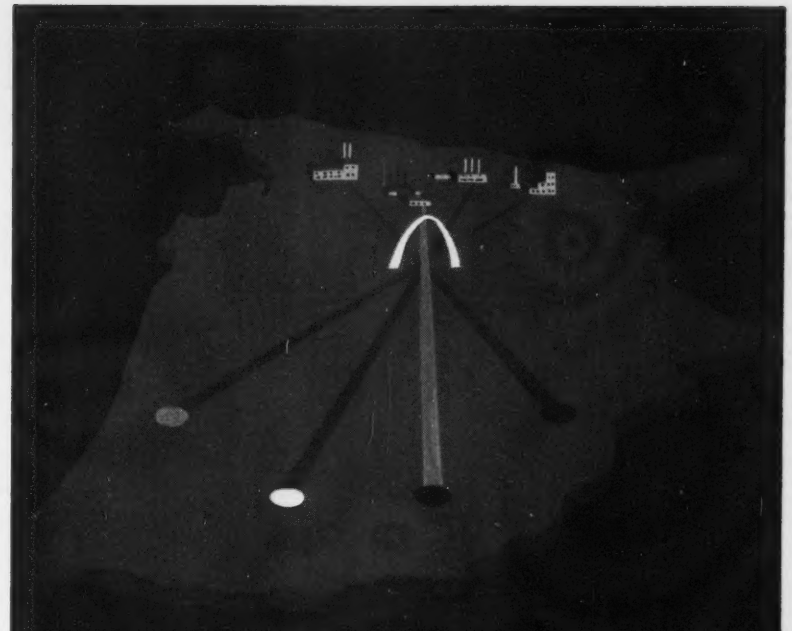
2.3.4, 3.2.2, 4.3.2, 6.2.5

Nitric-Hydrofluoric Acid Evaluation Test for Type 316L Stainless Steel. Donald Warren. Am. Soc. Testing Materials Bulletin, No. 230, 45-56 (1958) May.

The 10% nitric-3% hydrofluoric acid test can be successfully used to evaluate the intergranular corrosion resistance of type 316L stainless steel.—BTR. 16082

2.3.4, 3.2.2, 6.2.5

Intergranular Corrosion Resistance of Austenitic Stainless Steels. A Ferric Sulfate-Sulfuric Acid Test. M. A. Streicher, du Pont. Am. Soc. Testing Materials Bull, No. 229, 77-86 (1958) April.



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Characteristics of evaluation methods (boiling nitric acid test, copper sulfate-sulfuric acid test, nitric acid-hydrofluoric acid test and oxalic acid etch test) now in use are discussed. Various limitations inherent in each of these indicate need for new evaluation test. Development and performance of ferric sulfate-sulfuric acid test which fulfills requirements are described. Test is specific for intergranular attack associated with carbide precipitation in unstabilized steels. It takes only 120 hours, utilizes simple flask and condenser apparatus and permits testing of several specimens in same flask. Test solution consists of 600 ml boiling 50% sulfuric acid with an amount of ferric sulfate depending on number of specimens used per flask. About 10 g of ferric sulfate is required for each gram of steel dissolved. Total amount of ferric sulfate must not exceed solubility, 48 g per 600 ml at boiling temperature. There is practically no consumption of acid and no effect of corrosion products on rate of attack. Results are given of tests using new method on Types 304, 304L, 316 and 316L, and are compared with results of standard oxalic etch, standard nitric acid tests and, in some cases, nitric acid-hydrofluoric acid and copper sulfate-sulfuric acid tests. Proposal for evaluation sequence for austenitic stainless steels is outlined. Graphs show effects of sulfuric acid and ferric sulfate concentrations. Tables, photomicrographs.—INCO. 15943

2.3.7, 2.3.9

A New Approach to the Measurement of Coating Thickness by Fluorescent X-Ray Absorption. F. A. Achey and E. J. Serfass. Lehigh Univ. J. Electrochem. Soc., **105,** No. 4, 204-205 (1958) April.

Absorption of x-ray fluorescence from base metal of plated sample is most frequently used x-ray method for coating thickness determinations. If both coating and base metal yield fluorescence x-rays, it is necessary to isolate base metal fluorescence before measurement. Balanced filters are used for isolating fluorescence from base metal. Results show close agreement with crystal reflection techniques. Simplicity of method, together with low cost equipment and high accuracy, recommend it as useful tool in measuring plate thickness. Data are presented for iron coating on silver and on zirconium base metals.—INCO. 16037

2.3.9, 5.4.5

Modern Methods of Measurement in Paint Technology. G. Neumann. Farbe u. Lack, **64,** No. 1, 38 (1958).

Measurements of film thickness based on magnetic and capacitative principles, porosity measurements and viscosity and flow measurements were discussed. —RPI. 15488

2.3.9

Film Thickness Determination from Substrate X-ray Reflections. D. T. Keating and O. F. Kammerer. Brookhaven National Lab. Rev. Sci. Instruments, **29,** 34-36 (1958) January.

A method is given for the determination of the thickness of films using x-ray diffracted from the substrate. The x-rays pass through the film and are diffracted by the substrate back to the counter, the intensity being reduced by adsorption due to the double transmission through the film. No assumptions need be made about the substrate reflection. Unknown conditions of the substrate are eliminated by measurement of the intensity of two orders of a reflection, or measurement of the intensity of a reflection using two different incident radiations. The method is suitable for all types of film, and is particularly useful in measurements of films containing elements of the substrate for which cases x-ray fluorescence techniques are of little value. The procedure is illustrated with a measurement of the thickness of zirconium nitride on a zirconium substrate. (auth). —NSA. 15425

2.4 Instrumentation

2.4.2

An Accelerated Corrosion Test Chamber. W. Hess. Corrosion Prevention and Control, **5,** No. 5, 47-51 (1958) April.

Describes accelerated corrosion test chamber and test procedure developed at the Swiss PTT Chemical Laboratory. New testing method possesses advantage compared to principle of jets, namely, that air circulation is substantially reduced. Aerosol apparatus, which constitutes main part of new test chamber, permits material to be introduced without production of air currents. It is possible, simultaneously with aerosols, to introduce by very simple means an inert gas or chemically active gas as required, to produce all those conditions which are encountered in practice. Results are presented of tests on various paint coatings on iron plates. Table lists climatic conditions for five test sequences in the corrosion test chamber. —INCO. 15680

2.4.2, 3.5.8, 2.3.7

A High Temperature Vacuum and Controlled Environment Fatigue Tester. G. J. Danek, Jr. and M. R. Achter. U. S. Naval Research Lab., May, 1958, 12 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. (Order PB 131737).

Equipment was developed for reverse-bending fatigue tests at elevated temperatures in vacuum. Large strain amplitudes at low frequencies are used to produce failures in approximately 10^5 cycles. In addition to environmental studies, this equipment may be used for metallographic investigations of fatigue damage and crack initiation and growth at elevated temperatures. A major design problem, the transmission of motion through a vacuum seal, was circumvented by the use of a magnetic coupling driven at the resonant frequency of the specimen. Two methods of vibrating the specimen were developed. One uses two oscillating permanent magnets outside the vacuum chamber, while the other employs two electromagnets excited alternately. A thyratron circuit automatically stops the test when a crack develops. Fatigue data were gathered for type 316 stainless steel in a vacuum of 3×10^{-5}mm of mercury through a range of stresses at 1500 F.—OTS. 16033

2.4.3, 2.3.9, 7.6.4

Pinpointing Boiler-Tube Corrosion. R. M. Peterson. Magnaflux Corp. Power, **102,** No. 1, 112-113 (1958) Jan.

Discusses method of radiographic inspection in which an encapsulated radioactive isotope takes pictures of tube's insides and immediately pinpoints corrosion areas. Results of tests were highly satisfactory. Degree of corrosion showed plainly on film. Method can virtually eliminate forced boiler shutdown caused by tube corrosion. Photos. —INCO. 15476

3. CHARACTERISTIC CORROSION PHENOMENA

3.2 Forms

3.2.2, 6.3.6

Formation of Flakes on Copper Wires. (In German.) E. Lindau. Z. Erzbergbau u. Metallhüttenw., **10,** 504-509 (1957) October.

"Flakes" are the tiny copper particles adhering but not bonded to the surface of copper wires. To obtain quantitative estimate of flake formation a certain amount of wire was washed with trichlorethylene under ultrasonic excitation. Reasons for the flake formation were oxide formation during heating in oxidizing atmospheres; surface roughness of the wire during drawing; copper grades with oxide inclusions. 3 references.—MR. 15954

3.2.2, 6.3.15, 8.9.1, 3.5.9

High Temperature Brittleness in Titanium Alloys. N. Makrides and W. M. Baldwin, Jr. Case Institute of Technology. U. S. Wright Air Development Center, U. S. Air Force. June, 1957, 29 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington, D. C. (Order PB 13138.)

Purpose of this study was to determine whether titanium or its alloys will become brittle at high temperature and thus diminish their usefulness in aircraft structures. A review was made of various types of brittleness found at high temperatures in other metals and alloys so that any brittleness found in titanium could be compared and identified. Materials were an alpha titanium alloy and an alpha-beta titanium alloy. Specific studies were made of strain aging phenomena, high-temperature brittleness and stress-rupture behavior. —OTS. 15010

3.2.3, 5.9.2, 6.2.5

Pickling Stainless to Remove Scale. W. E. McFee. Armco Steel Corp. *Steel,* **141,** No. 22, 103-104, 107 (1957) Nov. 25.

Scale formed on stainless steels by exposure to high temperatures and furnace gases or from welding impairs corrosion resistance and is a hazard in fabricating unless it is properly removed. Scale adheres tightly to stainless and is more difficult to remove than that on carbon steel. Mechanical and chemical methods of scale removal are described. Seven pickling treatments are listed. Photos.—INCO. 14965

3.2.3, 6.2.2

Some Basic Problems of the Formation and Adherence of Scale on Iron. (In German.) H. Engell and F. Wever. Acta Metallurgica, **5,** 695-702 (1957) Dec.

Some kind of Kirkendall effect occurs due to preferential diffusion of either metal or oxygen in an oxide layer during the scaling of metals. If the metal diffusion predominates, voids are formed between scale and metal, or the oxide layer is porous. Both these phenomena influence the mechanical adherence of scale on the metal and the kinetics of oxidation. In the case of preferential diffusion of oxygen there will be a com-

pressed region at the boundary metal to oxide (if the oxide has a larger specific volume than the metal) which facilitates the peeling-off of the scale. 6 references.—MR. 15767

3.4 Chemical Effects

3.4.3, 8.4.5

Rates of Corrosion Product Removal from Circulating System by Film Formation and Settling. J. C. Suddath. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., CF-58-4-119, April 24, 1958, 5 pp. Available from Office of Technical Services, Washington, D. C.

Iron oxide deposits removed from high pressure system of HRT after the first power run will be analyzed for Fe^{59} in an attempt to determine the time these deposits were circulated prior to deposition (settling or film formation). Deposits available are film from corrosion specimens (stainless steel, zirconium and titanium) and settled particles. (auth)—NSA. 15970

3.4.3, 4.3.3

Effect of Ferric Oxide on the Process of Steel Oxidation in Alkali Solutions. (In Russian.) E. I. Levitina. *Zhur. Neorg. Khim. (J. Inorganic Chemistry)*, **2**, 1158-1161 (1957) May.

Study of the solubility of ferric oxide in alkali solutions and effect of its concentration on thickness of the magnesium oxide film formed.—BTR. 14790

3.4.3, 3.4.9, 5.3.4

White Rust on Galvanized Ware—Its Origin and Prevention. (In German.) K. Daeves. Draht, **8**, No. 8, 334-335 (1957) August.

Galvanized articles sometimes suffer very rapid corrosion, accompanied by the appearance of a loose white deposit consisting of basic zinc carbonate and zinc oxide. The common feature of all storage conditions favorable to white rust corrosion is the condensation of moisture. The attack can be induced by moistening the surface of the article with distilled water and keeping it in humid air. It can be avoided by storing and transporting the goods in well-aired containers or by greasing the surface. Chromate treatment does not ensure protection under all conditions.—ZDA. 15561

3.4.6, 6.3.20

The Oxidation of a Zirconium—50 w/o Uranium Alloy in Oxygen. L. D. Kirkbridge and D. E. Thomas. Westinghouse Electric Corp. U. S. Atomic Energy Commission Pubn., WAPD-LSR(MM)-43, Sept. 26, 1955 (Declassified March 21, 1957), 11 pp. Available from Office of Technical Services, Washington, D. C.

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The oxidation studies were made on uranium-zirconium specimens machined from strip stock which was previously hot rolled at 1450 F. Oxidized specimens were sectioned and examined by X-ray techniques. Corrosion data are included and the phases present as a function of distance from the oxide-gas interface are tabulated. Equations are given for the linear corrosion characteristics. The investigation showed that the oxidation product is a tetragonal structure below 400 C and that the effect of O on the E phase is pronounced from 750 to 900 C.—NSA. 15349

3.4.6, 3.2.3, 6.2.2

Speed of Scaling of Pure Iron in Oxygen at Diminished Pressures and in Carbon Dioxide. (In German.) Norbert G. Schmahl, Hans Baumann and Hermann Schenck. Archiv fur das Eisenhüttenw., **29,** 147-152 (1958) March.

Velocity of scaling of electrolytic iron at 950° and at varying pressures. A linear relationship is found at lower pressures, a parabolic at higher pressures. Annealing of iron in pure carbon dioxide at 950° produces fine crystalline scale; no definite conclusions can be given on scaling regularity. Velocity of scaling is greater with increasing oxygen pressure.—MR. 16121

3.4.8, 6.2.2

Causes of Rapid Rust Film Formation on Cast Iron. (In German.) G. Schikorr. Metalloberflache, **12,** 81-84 (1958) March.

Chlorine ions penetrate to graphite lamellas and remain there in spite of grinding. Depth of penetration corresponds to the length of lamellas. A 40% relative atmospheric humidity suffices to start rusting. Almost 200 microns at superficial layer must be removed to prevent rust deposition.—MR. 15477

3.4.8, 6.2.5

The Effect of Fluoride on the Corrosion of Stainless Steel in the Presence of Excess AL^{+3} Ion. Arnold R. Olsen. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., ORNL-947, March 27, 1951 (Declassified Feb. 7, 1957), 9 pp. Available from Office of Technical Services, Washington, D. C.

Corrosion tests of Type 347 stainless steel for two weeks in boiling uranyl nitrate, sulfuric acid media, with and without additions of Al^{+3} and F^{-1} in a 2:1 mole ratio, were made. The results show that although the presence of aluminum greatly reduces the deleterious effect of fluoride, the corrosion rates are still three to four times greater than when no fluoride is present. (auth.)—NSA. 15877

3.4.8, 3.6.5, 3.8.4, 6.2.2

A Study of the Effect of Chloride Ion on Films Formed on Iron in Sodium Nitrite Solutions. G. W. Mellors, M. Cohen and A. F. Beck. National Research Council, Ottawa, Canada. J. Electrochem. Soc., **105,** No. 6, 332-338 (1958) June.

The effect of chloride ion on the potential of iron in sodium nitrite solutions has been measured. This has been related to the surface composition and topography by the techniques of electron diffraction and electron microscopy. The effect of initial surface preparation has also been investigated.

It is shown that in the absence of chloride ion the potential is noble and steady and that the surface remains practically unchanged. A film of δ-ferric oxide is shown to be present by electron diffraction. When chloride ion is added to the system the potential becomes unsteady and less noble. Inclusions of a second phase grow in the oxide layer. This is shown to be $\delta\text{-}Fe_2O_3 \cdot H_2O$ or lepidocrocite. The lower and unsteady potential is probably due to the decrease in resistance of the anodic areas, which correspond to the inclusions in the oxide layer. 16083

3.4.8, 4.3.2, 6.2.5

The Effect of Chromium in Nitric Acid Solution on the Corrosion Resistance of Types 304L and 309SCb Stainless Steel. Kenneth L. Sanborn. Hanford Atomic Products Operation. U. S. Atomic Energy Commission Pubn., HW-32877, August 31, 1954 (Changed from Official Use Only January 28, 1957), 6 pp. Available from Office of Technical Services, Washington, D. C.

The corrosive effects of Cr^{3+} and Cr^{6+} in nitric acid solutions of various concentrations on types 304L and 309SCb austenitic stainless steel are presented. —NSA. 15420

3.4.8, 3.4.7, 6.4.2

Uniform Aqueous Corrosion of Aluminum—Effects of Various Ions. Vi. H. Troutner. Hanford Atomic Products Operation. U. S. Atomic Energy Comm., HW-50133, June 10, 1957, 52 pp.

Most important variable in water quality on uniform corrosion of aluminum was pH. Solutions of distilled water containing phosphate ions and citrate ions were only media which showed a definite specific ion effect. Phosphate inhibited corrosion and citrate increased it. Oxalate ions appeared to increase corrosion, but effect is not definitely demonstrated by data. Corrosion was dependent only on pH in tap water, reactor process water, distilled water and distilled water containing up to 100 ppm of chloride, nitrate, sulfate, bicarbonate, hydrogen peroxide, acetate, arsenate, silicate, dichromate, molybdate and mixtures of these ions. Solutions of up to 10,000 ppm chloride in distilled water showed no specific ion effect upon uniform aluminum corrosion. Effects of 0-10 ppm of dichromate and phosphate ions were investigated over pH range 4-7. Phosphate at 10 ppm inhibited corrosion in solutions containing up to 2 ppm of dichromate, but at 10 ppm dichromate the phosphate does not appear to inhibit corrosion. Also at 10 ppm dichromate, effect of pH is diminished over pH range 4-7. Tables.—INCO. 15127

3.4.8

Effect of Sulphur Content on Blast Furnace Slag and Its Corrosion Behavior. (In German.) Hans Ernst Schwiete, Ludwig Zagar, Peter Dickens and Paul Konig. *Arch. Eisenhuttenw.,* **28,** 187-194 (1957) April.

Corrosion tests for various conditions; surface treatment of fiber slag; effect of sulfide ion on corrosion of sheet iron. —MR. 15070

3.4.8, 8.4.3, 3.5.9

High Temperature Hydrogen Sulfide Corrosion in Commercial Sovaformer Units. Pt. 2. E. B. Backensto, R. D. Drew and J. N. Vlachos. Corrosion Technology, **5,** 53-56 (1958) Feb.

Test results obtained in two commercial sovaformers show that both temperature and hydrogen sulfide are basic variables in this type of corrosion. Aluminizing provides protection from sulfide corrosion attack. Use of 18-8 chromium-nickel alloys is also suggested.—MR. 15974

3.4.8, 6.3.6, 7.6.8

Corrosion of Distillery Equipment. (In German.) Dietmar Plottner. Werkstoffe u Korrosion, **9,** 261-262 (1958) May.

Copper is especially sensitive to hydrogen sulfide and a considerable quantity of grayish black copper sulfide accumulates in the receptable of a copper still.—MR. 16116

3.4.9, 2.2.3

Corrosion of Steel in Moist Air. J. T. Crennell. J. Applied Chem., **8,** Pt. 4, 270-272 (1958) April.

Specimens of steel stored at room temperature and approximately 100% relative humidity for periods up to 7 years have shown a remarkable absence of corrosion. Observation was made in Portsmouth Dockyard, where industrial and marine conditions are combined, in a chemical laboratory where casual fumes are likely to contaminate the air, as the specimens were kept in vessels which were repeatedly opened. Photos. —INCO. 15632

3.5 Physical and Mechanical Effects

3.5.3

Phenomenon of Cavitation. (In Italian.) L. C. Burrill. *Tecnica Italiana,* **21,** No. 2, 95-103 (1956) March.

Physical nature, effects of localized erosion and vibrations and noises caused by cavitation. Some recent experiences and methods of avoiding cavitation. Photographs, diagrams.—BTR. 12396

3.5.3

On the Erosion of Metal Surfaces by Ultrasonic Cavitation. (In German.) R. Esche and H. Kimmel. *Z. Naturforsch.,* **11a,** 514-515 (1956) June. 13596

3.5.3, 2.3.7, 8.9.1

Rain Erosion: Barrier to High Speed Flight. N. E. Wahl. *Research Trends, Cornell Aeronaut. Lab.,* **4,** No. 3, 5-7 (1956) Fall.

Erosion by water-drop impact has become an increasingly important research problem in the post-war era of high speed aircraft. Plastics used in noses of bombers and in radomes erode completely below 400 miles per hour. Description of test equipment and tests on materails including stainless steel, chromium and nickel base alloys is given. Each type of material has a threshold velocity below which erosion is not initiated even after exposure as long as 24 hours. This critical velocity is proportionate to tensile strength and hardness of materials. Elastomers such as neoprene have the greatest resistance to rain erosion of any non-metallic materials and were used as a starting point for successful development of an electrically conductive coating now universally used on high speed military aircraft. Photos. —INCO. 13732

3.5.3, 5.3.4

Cavitation Resistance of Electrodeposited Chromium. M. G. Timerbulatov and V. E. Khromov. Vestnik Mashinostroeniya, **38,** No. 2, 56-58 (1958). Translation available from Henry Brutcher, Technical Translations, P. O. Box 157, Altadena, California.

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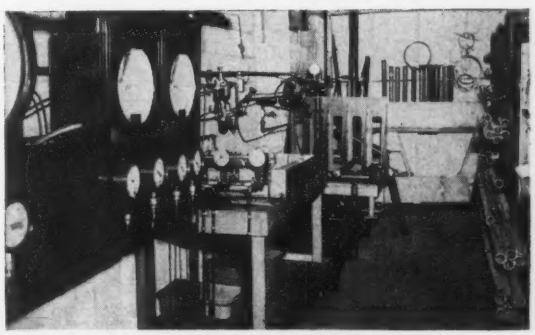

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Experiments on cavitation resistance of chromium plates deposited under various conditions. Index of cavitation resistance: Loss of weight of specimens exposed to vibration with a magnetostrictional vibrator in a liquid. Setup for plating the specimens. Lack of correlation between cavitation resistance and Vickers hardness of chromium plates. Factors involved in cavitation resistance such as type of plate (dull vs. bright); thickness, porosity, brittleness and residual stress state of plate; and hardness of basis metal. Results—cavitation resistance improved by chromium deposits. 6 figures.—HB. 15647

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3.5.3, 8.9.1, 7.3

Study of Damaging Effects of Cavitation Erosion to Ships' Underwater Structures. J. Z. Lichtman, D. H. Kallas, C. K. Chatten and E. P. Cochran, Jr. Paper before Am. Soc. Mechanical Engrs., Annual Mtg., New York, December 1-6, 1957. ASME Paper No. 57-A-156, 1958, 24 pp.

Although fluid cavitation may occur adjacent to many underwater appendages of vessels at normal operating speeds, there is considerable evidence that areas in which cavity collapse is the primary cause of damage are restricted to propellers. Damage to ships' struts, rudders and other appendages is shown to be primarily corrosive in nature aggravated by mechanical scouring action. Neither aluminum nor plastic structural materials such as polyvinylchloride and polyester materials are as resistant to cavitation-erosion damage as brass, mild steel, stainless steel, manganese bronze and ballistic steel, latter group increasing in resistance in order indicated. Organic coatings have afforded good anticorrosive protection to propeller shafts and fair protection to struts and rudders in service. Table showing cavitational erosion resistance of sprayed metallic coatings includes nickel and Monel. Photos, diagrams, 32 references.—INCO. 15929

3.5.4, 6.3.21

Proton Irradiation Effects in Thorium. CHARLES J. MEECHAN. North American Aviation, Inc. U. S. Atomic Energy Comm. Pubn., NAA-SR-1079, Dec. 15, 1954 (Declassified March 5, 1957), 16 pp. Available from Office of Technical Services, Washington, D. C.

Iodide-processed thorium foils were irradiated with 9-Mev protons at temperatures below —140 C. The recovery of electrical resistance upon annealing was studied in the range 0 to 75 C where tempering curves showed rapid changes taking place. Determinations of the activation energy associated with this process were made and the mean value obtained was 1.22 ev. Correlations of this result have been made with those found previously for copper. From these comparisons, a tentative assignment of the motion of interstitial atoms in thorium has been made for this process. In addition, some evidence has been found which illustrates the corrosive action that water has on thorium at temperatures as low as 0 C. (auth)—NSA. 1479

Corrosion Tests (on Aluminium and Its Alloys) in the Presence of Radioactive Emanation. K. Lintner, E. Nachtigall and E. Schmid. Metall, **11,** No. 1, 31-35 (1957) Jan.

The presence of radioactivity increases the corrosion rate of 99.5% aluminum, wrought aluminum-manganese, aluminum-3% magnesium, aluminum-copper-magnesium, aluminum-magnesium-silicon, aluminum-zinc-magnesium and cast-aluminum-12% silicon alloys, in air, water, water vapor and 3% sodium chloride solution; in addition, it reduces the mechanical strength of many of the materials. The effect may be due to one or more of the following causes: (1) radioactivity promoting hydrogen peroxide formation and thereby accelerating corrosion; (2) α particles penetrating the oxide skin, thus rendering it porous to the corrosive medium; and (3) α particles impinging on the metal and producing local hot spots leading to the formation of local cells.—MA. 15831

3.5.4, 3.8.4, 6.4.2

Photo Effects with Anodic Oxide Layers on Tantalum and Aluminum. W. Ch. van Geel, C. A. Pistorius and P. Winkel, Philips Research Rept., **13,** No. 3, 265-276 (1958) June.

Deals with photoelectric properties of the system aluminum/aluminum oxide/electrolyte during irradiation with ultraviolet light. The electrolyte has also been replaced by a transparent layer of a metal or a semi-conductor. When the system is short-circuited, a photocurrent (i_o) is produced which is proportional to the intensity of irradiation. With a high external resistance a photo emf (V) appears which shows considerable inertia as a function of the time t and which can be written as $V = V_{max}(1 - e^{-kt})$. It appears that k is proportional to the intensity of irradiation. The value of k decreases with increasing thickness (d) of the layer. After correcting for this change of k, which is probably due to absorption, it is found that $i_o d$ = a constant. An attempt is made to explain the observed phenomena by assuming that the work function aluminum(aluminum oxide is smaller than the work function electrolytes/aluminum oxide.—ALL. 16103

3.5.4, 8.4.5

Feasibility Study of a Dynamic Pressurized Water Irradiation Facility in a Vertical Hydraulic Rabbit Facility at the Materials Testing Reactor. WALTER SCHWARTZ. Westinghouse Electric Corp. U. S. Atomic Energy Commission Pubn., WAPD-PWR-CP-1866, Feb. 29, 1956 (Declassified March 21, 1957), 13 pp. Available from Office of Technical Services, Washington, D. C.

This facility was evaluated for its utilization to study the combined effects of irradiation and corrosion on defectively clad fuel elements. The design is such that these effects may be studied during steady-state irradiation and simulated power cycling.NSA. 15076

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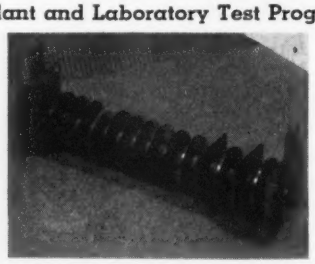

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3.5.4, 3.7.3, 6.2.2, 6.2.5, 6.3.1

Radiation Effects on Welds and Notches in Plain Carbon Steels, Stainless Steels, and Non-Ferrous Alloys. D. O. LEESER. Paper before Am. Soc. Testing Materials 2nd Pacific Area Nat'l Mtg., Los Angeles, Sept. 20, 1956. ASTM Special Technical Pubn. No. 208, Symposium on Radiation Effects on Materials, 1, 154-161 (1957) July.

Materials tested included stainless steels, nickel, Monels, Inconel, Hastelloy, Stainless W, 17-4 PH, Stellites and zirconium. Austenitic stainless steels, 18-8 types and certain non-ferrous alloys offer advantage of higher initial ductility and lack of entirely brittle failures. Irradiation effects can be expected to be less severe on stainless steel reactor vessels operating at relatively higher temperatures than carbon steel vessels operating at lower temperatures. Criteria for evaluation include stress, strain, strain rate, irradiation dosage and time and temperature in service. Embrittlement under irradiation may occur in carbon steels unless a sufficiently high protective temperature is used. Austenitic stainless steels are not seriously embrittled by irradiation since nonductile-to-brittle transition temperature is encountered. All fracture surfaces appeared ductile even after irradiation irrespective of testing method or of irradiation temperature. Photographs, tables.—INCO. 15051

3.5.4, 6.3.17

Effects of Irradiation on Corrosion Resistance of Some High Uranium Alloys. S. GREENBERG AND J. E. DRALEY. Argonne National Lab. *Nuclear Science & Eng.*, **3**, No. 1, 19-28 (1958) Jan.

Three corrosion resistant uranium base alloys corrosion-tested in high temperature water were: Uranium-5 zirconium-1.5 niobium, uranium-3 niobium-0.5 tin and uranium-3.8 silicon. High temperature corrosion resistance of metastable alpha alloys was destroyed when irradiated to burnup exceeding 0.1 at .%; this confirms earlier data. Uranium-3.8 silicon alloy retains corrosion resistance after irradiation to much greater degree than do metastable alpha alloys. Proper heat treatment of assembly consisting of uranium-2 zirconium core and Zircaloy-2 cladding produces structure which remains corrosion resistant in presence of small defects. Corrosion resistance of diffusion bond between uranium-2 zirconium and Zircaloy-2 was not adversely affected by irradiation to low burnup (0.1 at .%). Corrosion rates are given in mg/cm²/day. Tables.—INCO. 15169

3.5.4, 8.4.5, 3.7.3

Radiation Effects in Pressure Vessel Materials and Welds. J. C. WILSON. Oak Ridge National Lab. Paper Presented at Meeting of AEC Welding Committee on October 28, 1954 at Cincinnati, Ohio. U. S. Atomic Energy Commission Pubn., CF-54-10-102 (Del.), Nov. 15, 1954 (Declassified March 12, 1957), 23 pp. Available from Office of Technical Services, Washington, D. C.

Radiation-induced changes in the physical and mechanical properties of reactor structural metals are discussed. Emphasis is placed on pressure vesel material and welds. A theory is presented to explain some of the observed property changes. Data are included from studies on the effects of irradiation on aluminum alloys, carbon steel, nickel alloys, stainless steel and carbon steel welds.—NSA. 15148

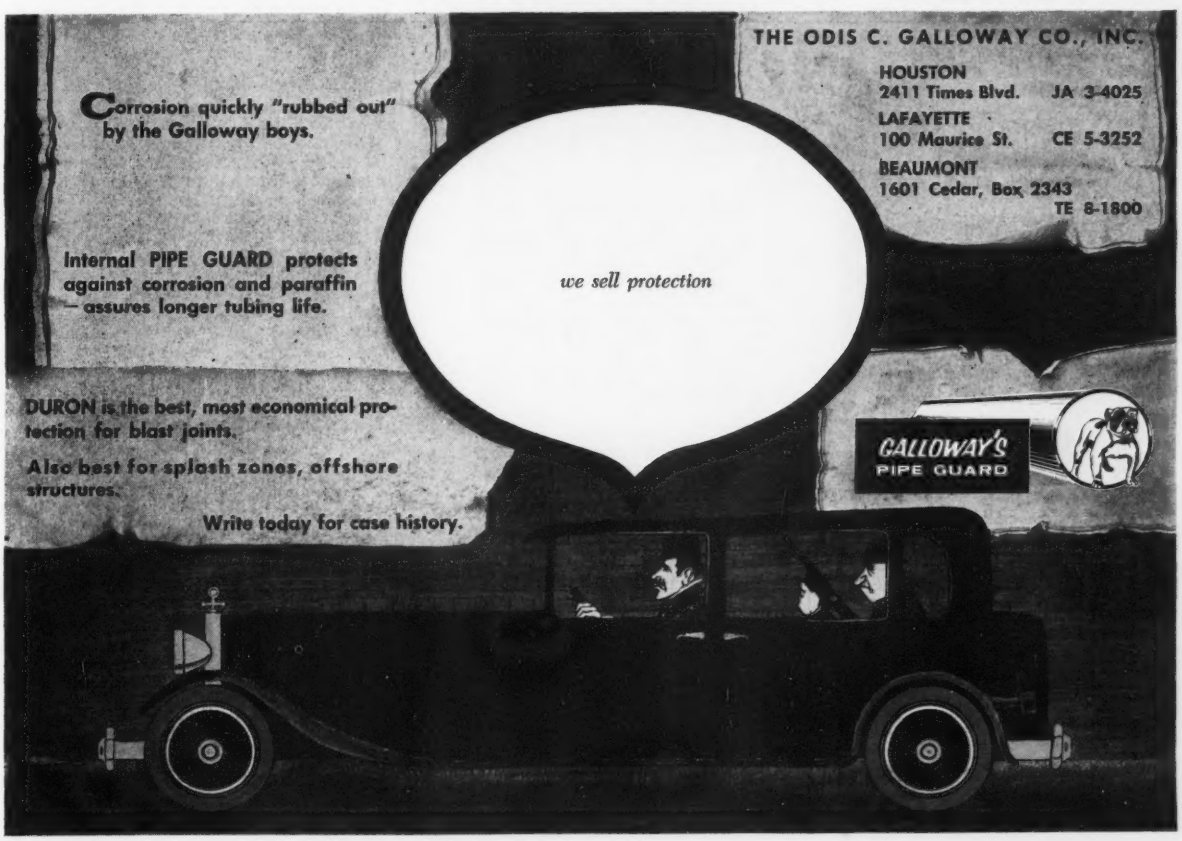
3.5.4, 8.4.5, 3.4.3, 6.2.5

Review of the Corrosion Product Radioactivity Program at the Army Package Power Reactor. A. L. Medin. Alco Products. U. S. Atomic Energy Commission Pubn., APAE-31, April 25, 1958, 19 pp. Available from Office of Technical Services, Washington, D. C.

An analysis and a summary are given of the radioactivity buildup program at the Army Package Power Reactor. Due to the wide fluctuation of water and crud results, only general interpretations can be made with this data. Metal test coupon data indicate a substantially greater buildup on Croloy 16-1 metal than on Type 304 stainless steel. Coupled with the decreased ability to remove the radioactivity buildup on Croloy 16-1 by conventional descaling techniques, the implication is that this metal might pose a serious problem for use as steam generator material. It was emphasized, however, that results to date are only preliminary and extensive additional experimentation would be required to reach more definite conclusion. (auth.) —NSA. 16011

3.5.4, 8.4.5

Some Radiation Chemistry Aspects of the Corrosion of Metals on Irradiation. W. L. Primak and L. H. Fuchs. Argonne National Lab. U. S. Atomic Energy Commission Pubn., ANL-4763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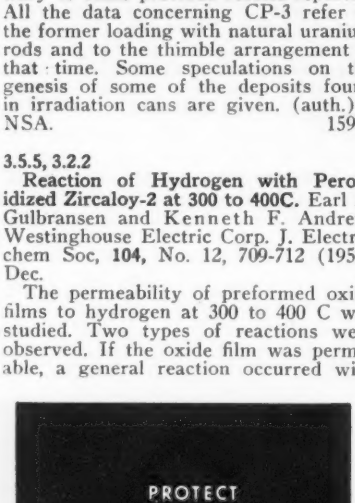
(Del), April 21, 1952 (Declassified with Deletions March 14, 1957), 23 pp. Available from Office of Technical Services, Washington, D. C.

Certain metals and alloys (e.g., nickel, cobalt, lead, aluminum, inconel) which are subject to nitric acid corrosion were found to form nitrates and basic nitrates when irradiated in moist air in reactors. Lead was found to form oxides when irradiated in moist oxygen or in water vapor. Some of the corrosion products appear as deposits in unexpected portions of the irradiation containers. The salts formed on the metals may lead to an unexpected spread of radioactivity. The corrosion may proceed to the point of producing serious damage to samples and parts in a nuclear reactor. The rate of nitrogen fixation was obtained in two reactors, CP-3 and X-10, and the yields calculated seem reasonable for the respective conditions of irradiation, contrary to some previous results reported. All the data concerning CP-3 refer to the former loading with natural uranium rods and to the thimble arrangement at that time. Some speculations on the genesis of some of the deposits found in irradiation cans are given. (auth.)—NSA. 15913

3.5.5, 3.2.2

Reaction of Hydrogen with Peroxidized Zircaloy-2 at 300 to 400C. Earl A. Gulbransen and Kenneth F. Andrew. Westinghouse Electric Corp. J. Electrochem Soc, **104,** No. 12, 709-712 (1957) Dec.

The permeability of preformed oxide films to hydrogen at 300 to 400 C was studied. Two types of reactions were observed. If the oxide film was permeable, a general reaction occurred with the formation of zirconium hydride phases on the surface. If the oxide film was relatively impermeable, reaction occurred at the edges of the sample where defects probably exist in the oxide. The reaction starts at localized areas and spreads through the metal with hydride and oxide film spalling from the metal continually. (auth.)—NSA. 15851



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3.5.8, 7.10

Fatigue Strength of Bolts Reduced by Longitudinal Flaws. J. Viglione. Naval Air Experimental Station. *Product Eng.,* **28,** No. 3, 203-205 (1957) March.

To determine exact influence of longitudinal flaws in reducing service life of bolts, fatigue tests were made on nickel-chromium-molybdenum steel (8630, 8375 and 8740) bolts in which longitudinal flaws had been detected by circular magnetization in magnetic particle inspection. All fatigue failures were in threads and related to flaws. Photomicrographs. —INCO. 13845

3.5.8, 7.6.4, 4.6.2

Caustic Cracking: Its Occurrence in Steam Boilers. D. D. Jones. Paper before Soc. Chem. Ind., Corrosion Group, 1956. *Corrosion Technology,* **4,** No. 2, 56-59 (1957) Feb.

Caustic cracking occurs in steam boilers at 150-300 psi. Sodium hydroxide, added to feed water for scale prevention, becomes concentrated in seams, rivet crannies and other fissures until concentration high enough to produce corrosion is reached. Where steel is in state of tension attack is intergranular. Cracks may run from one rivet hole to another. Causal theories and probable mechanism of caustic cracking are considered. In experiments at National Physical Laboratory, no cracks formed in boiler specimens under tension for 5 years in absence of corrosive attack; no intergranular attack was suffered by steel stretched beyond yield point in sodium hydroxide at 225 C but early breakage occurred; while at temperatures up to 470 C and under pressure, intergranular attack accompanied by decarburization occurred. At 410 C intergranular cracking only occurred in cold-worked steel. Black oxide on metal at relatively low temperatures was ferric oxide, but magnetite appeared at higher temperatures. Magnetite acts as cathode in concentration cell. Hydrogen type of attack seems to produce cracking only in cold-worked steel whereas oxide type is dangerous even with annealed material. Prevention of caustic cracking by various methods is considered and effect of steel quality is discussed.—INCO. 13907

3.5.8

A Theory of the Origin of Fatigue Cracks. N. F. Mott. Acta Metallurgica, **6,** No. 3, 195-197 (1958) March.

A model based on the concept of cross slip is given to explain how a slip band can develop into a crack if dislocations in the band are free to move backwards and forwards.—ALL. 15592

3.5.8, 3.5.9, 3.7.3

Thermal Stress Problems in Practice. R. U. Blaser. Paper before Soc. Exptl. Stress Anal., Spring Mtg., Panel on Thermal Stresses, Pittsburgh, May, 1956. Proc. Soc. Exptl. Stress Anal., **15,** No. 2, 131-142 (1958).

Thermal stresses can occur from temperature differences within piece of material, from parts of assembled structure at different temperatures, or from combinations of materials having different thermal expansion characteristics. Examples of thermal stress problems arising in actual practice are discussed; problems are largely chosen from equipment in field of heat transfer and other high temperature applications. Welding, heat transfer and quenching stresses, dissimilar metal welds, related phenomenon of stress corrosion, and effects of thermal stresses on mechanical joints are topics covered.—INCO. 16122

3.5.8, 5.9.3, 6.2.3

Stress Corrosion of Mild Steel. Pt. II: The Effect of Shotblasting on the Corrosion-Fatigue of Mild Steel. (In Japanese.) Y. Minami. K. Inami and N. Okano. *Trans. Shipbuilding Assoc., Japan,* No. 102, 133-138 (1957).

Fatigue properties of as-rolled and shot-blasted mild steel plates were investigated in air, in sea water and under impressed current cathodic protection. By shotblasting, the fatigue limit in air rises about 20% over that of as-rolled plate. The reasons why the S-log N diagrams of two surface conditions are close to each other in the corrosion fatigue test and the fatigue limits under cathodic protection are lower than those in air were considered.—JSPS. 15373

3.5.9, 3.8.4, 6.2.1

High Temperature Oxidation of Iron-Nickel Alloys. Metallurgy Report No. 12. M. J. Brabers and C. E. Birchenall. Forrestal Research Center, Princeton Univ. U. S. Air Force Office of Scientific Research, Tech. Note 57-150, March 25, 1957, 23 pp.

The iron-rich corner of the phase diagram for iron-nickel-oxygen was determined at 1050 C. Great care was taken to assure equilibration which was not completely achieved in an earlier study. The mechanism of oxidation of iron-nickel alloys is discussed and a protective mechanism is described which differs somewhat from those suggested for other alloys. (auth)—NSA. 14226

3.5.9, 4.3.3, 3.4.3

High Temperature Corrosion of Metals by Vanadium Pentoxide. II. Behavior of Some Metals (Iron, Cobalt, Nickel, Chromium) and of Their Alloys. (In Italian.) A. Burdese and C. Brisi. *Metallurgia Italiana,* **48,** No. 8, 349-357 (1956) August.

Identification of the corrosion products formed on iron, cobalt, nickel, chromium and their alloys in the presence of vanadium pentoxide.—BNF. 13980

3.5.9, 6.2.1

High Temperature Oxidation of Iron-Nickel Alloys. M. J. Brabers and C. E. Birchenall. Princeton Univ. Metallurgy Rept. No. 12, March 25, 1957, 22 pp.; Air Force Office of Scientific Research, Tech. Note 57-150.

Determination of iron rich corner of phase diagram for iron-nickel-oxygen at 1050 C. Mechanism of oxidation of iron-nickel alloys is discussed and a protective mechanism is described which differs somewhat from those suggested for other alloys. Photomicrographs, tables, graphs, 18 references.—INCO. 14056

3.5.9, 6.3.10

Oxidation of Nickel-Chromium Alloys at High Temperatures. (In French.) J. Moreau. *Corrosion et Anticorrosion,* **4,** No. 211-16 (1956) June.

Scaling of nickel-chromium alloys (4.6, 11.3 and 19.8% chromium) and pure nickel in air at atmospheric pressure and 900-1300 C was studied. Investigation of

the structure of the alloys and surface films by X-ray diffraction and microscopy.—BNF. 13682

3.5.9, 6.1

Some New Materials for Missile and Power-Plant Applications. A. V. LEVY. *Aircraft Eng.*, **28**, No. 332, 357-361 (1956) Oct.

Mechanical properties and fabrication characteristics are considered for four materials developed for elevated temperature operation: reinforced plastics, titaninm alloys (titanium-5 aluminum-2.5 tin, titanium-6 aluminum-4 vanadium), molybdenum alloy (molybednum-0.5 titanium) and magnesium alloy (magnesium-3 thorium-0.75 zirconium). The last is dealt with most fully (material quality, corrosion protection, ramjet engine application, working sheet metal, welding and heat treating). Refractory coatings also mentioned.—BNF. 13635

3.5.9

Heat-Resisting Materials. W. NORDEN AND G. R. BARROW. Paper to Intern. Congr. on Materials for Aircraft Guided Missiles, 1955. *Metaux: Corrosion-Industries*, **31**, 488-499 (1956 Dec.

Review of compositions and properties (including creep) of standard heat-resisting alloys, with particular reference to vacuum-melted Hastelloy R-235.—INCO. 13676

3.5.9, 6.2.5

High Temperature Oxidation Characteristics of Some Manganese Steels. Part II. VED PRAKASH AND A. A. KRISHNAN. *J. Sci. Ind. Research*, **15B**, 600-607 (1956) October.

Metallography of the oxides formed at 600 and 700 C, effect of temperature on oxidation of manganese-aluminum steel, influence of composition. 10 references.—MR. 13662

3.5.9, 8.9.5, 2.3.7

Low-Temperature Brittleness. C. S. BARRETT. 1956 Edward De Mille Campbell Memorial Lecture before Am. Soc. Metals, Cleveland, October 10, 1956. *Metal Progress*, **70**, No. 6, 68-72 (1956) Dec.

Reviews scientific investigations of metallurgical behavior at low temperature which have shown the interrelation between brittle fracture of ship plate and many of the properties determined by crystal imperfections. Even with perfect welds and good design to eliminate stress concentrators, it was shown that notches could not be avoided throughout life of ship. This indicated need for material that is tough in presence of notches under service conditions. Use of 15-ft-lb. V-notch Charpy transition temperature to evaluate ductile-brittle behavior is discussed. Recent statistical analysis of transition temperatures in World War II and post-war ship plates indicates that service failure should be eliminated by use of steel with mean V-notch Charpy 15-ft-lb. transition temperature of 10 F or lower. Metallurgical variables which produce lower transition temperature are summarized. Application of low temperature testing is considered. Graphs.—INCO. 13501

3.7 Metallurgical Effects

3.7.2, 6.4.2

Development of a Corrosion Resistant Magnesium Alloy. Part I. Development of Magnesium Alloys for Better Corrosion Resistance. M. BALICKI, D. D'ANTONIO AND A. KRAVIC. Polytechnic Institute of Brooklyn. Wright Air Development Center, U. S. Air Force. August, 1957, 38 pp. Available from Office of Technical Services, U. S. Department of Commerce, Washington 25, D. C. (Order PB 131443).

A feasibility study was made of a novel scheme to improve corrosion resistance of magnesium by alloying with elements with lower surface tensions. These were expected to migrate to the surface and alter the corrosion behavior of the base metal. A number of elements having lower surface tension were added to magnesium to form very pure, dilute, binary alloys. Preliminary results indicated that alloys containing antimony, germanium, indium, tin and cadmium improve corrosion resistance. In salt spray tests, these alloys compared favorably with pure magnesium.—OTS. 15185

3.7.2, 6.4.2

Influence of Silicon Contained in Excess of the Ratio of Magnesium Silicide in Aluminum-Magnesium-Silicon Alloys. HAJIME YAMADA. *Light Metals* (Japan), No. 25, 45-50 (1957) July.

The effect of excessive silicon in the aluminum-magnesium-silicon alloys, containing more silicon than the ratio of magnesium silicide, on age-hardening, mechanical properties, corrosion and stress-corrosion resistance was studied and the following results were obtained.

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(1) The excessive silicon raised the hardenability of ageing of the alloy and reduced the ageing time. (2) Silicon contained in aluminum-magnesium-silicon alloy-T_6 in the ratio greater than magnesium silicide had an effect on tensile strength of the alloy. It was remarkable for the alloy containing 0.65% magnesium silicide solution heat treated at 520 C. The tensile strength decreased with an increase in the amount of magnesium silicide. Elongation was unchanged except a slight decrease for alloys containing excessive silicon up to 0.2%. When solution heat treated at 560 C, the alloys containing 1.0% magnesium silicide showed remarkable increase in tensile strength and the excessive silicon, over 0.5%, had the same effect for the alloys containing 0.65% magnesium silicide. Elongation decreased with an increase of excessive silicon. (3) Silicon in O-alloy in the greater ratio than magnesium silicide in O-alloy increased tensile strength and elongation. Increase of elongation of the alloy containing 0.65% magnesium silicide was striking. The maximum elongation was observed at 0.6% of excessive silicon and elongation decreased with an increase of excessive silicon more than 0.6%. (4) Electric resistance was increased slightly by the excess silicon. The change was not so remarkable when the excessive silicon was up to 0.2% for the alloys of 0.65% and 1.0% magnesium silicide. (5) Corrosion resistance decreased with an increase of excessive silicon. (6) Susceptibility to stress corrosion increased with an increase of excessive silicon but it is not so remarkable as the effect by the amount of magnesium silicide. The effect of excessive silicon would be remarkable for such alloy containing small amount of magnesium silicide as A 51S. The amount of silicon should be decided by the amount of magnesium in the alloy. Generally speaking, at least 0.2% more silicon than the ratio of magnesium silicide is recommendable for such low magnesium alloy as aluminum-magnesium (0.45 ~ 0.6%)-silicon alloy. (auth.) —ALL. 15101

3.7.2, 4.3.2, 6.3.17

Effects of Carbon Content on the Rate of Dissolution of Dingot Uranium in Nitric Acid. A. L. Bement and J. L. Swanson. Hanford Atomic Products Operation. U. S. Atomic Energy Commission Pubn., HW-52430, November 11, 1957, 20 pp. Available from Office of Technical Services, Washington 25, D. C.

Carbon additions made to dingot uranium by vacuum casting with a high-frequency electronic heater varied from 150 to 1500 ppm. The specimens were beta heat treated, then dissolved in nitric acid. Dissolution rates were determined for the linear, initial portion of the weight-loss-versus-time curves. The rates so obtained were compared with those for samples of ingot, and normal and high-carbon dingot uranium. The effect of grain size on the dissolution rate can be expressed as a power function.—MR. 16143

3.7.3

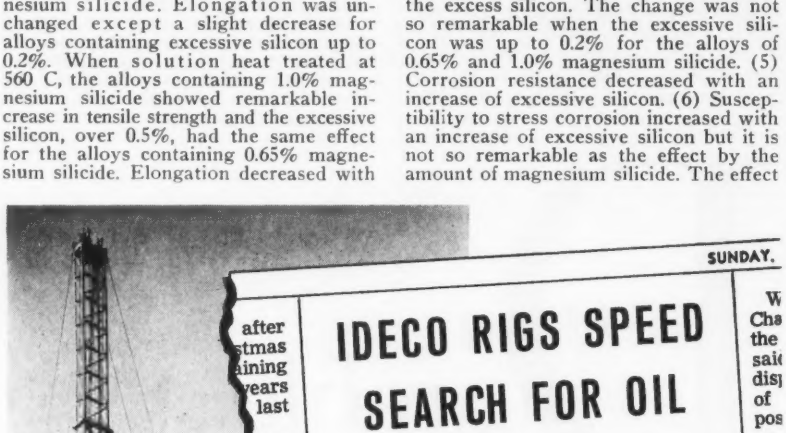
The Influence of Surface Roughness on the Fatigue Strength of Steels and Non-Ferrous Alloys. E. Siebel and M. Gaier. *Z. Ver. deut. Ing.*, **98**, No. 30, 1715-1723 (1956) October 21. Summary: *Engrs' Digest*, **18**, No. 3, 109-112 (1957) March.

From series of steels (including 18 chromium-9.5 nickel stainless) and nonferrous alloys (brass, aluminum-magnesium, aluminum-copper and magnesium) in specified heat-treated condition, fatigue-test specimens were machined to specified roughness and were tested in push-pull, bending, tension and torsion. Design formulae for various machining processes are established from test results. Limiting surface finish exists beyond which fatigue strength does not improve. Surface hardening treatment (nitriding) after machining tends to eliminate influence of surface roughness on fatigue strength.—INCO. 14097

3.7.3, 6.3.15

The Determination of the Effect of Heat Treatment on the Elevated Temperature Stress-Stability of Titanium Alloys. G. A. Lenning, M. L. Greenlee, W. M. Parris and H. D. Kessler. Titanium Metals Corporation of America for Wright Air Development Center, U. S. Air Force, Feb., 1958, 87 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. (Order PB 131724).

Many commercial titanium alloys can be heat-treated to high strength and thus offer promise of meeting the increasing temperature and thermal requirements of advanced aircraft. This study was undertaken to determine the effects of heat treatment on the stress stability or notch stability of three titanium alloys representative of the alpha-beta type. They were Titanium-140A (titanium-2 iron-2 chromium-2 molybdenum), Titanium-155A (titanium-5 aluminum-1.5 iron-1.5 chromium-1.5 molybdenum), and titanium-6 aluminum-4 vanadium. Duplex solution and age-type heat treatments were shown to provide improvement in strength and



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creep resistance of the alloys without loss of either stress stability or notch ductility. No significant differences between long time-low temperature versus short time-high temperature aging treatments were observed. The beneficial effect of heat treatment on creep resistance was found to decrease markedly at higher exposure temperatures, but was significant for conditions which would include maximum useful temp. of alloys.—OTS. 15691

3.7.3, 3.8.4

The Influence of Surface Films on the Pressure Welding of Metals. R. F. Tylecote, D. Howd and J. E. Furmidge. Brit. Welding J., **5**, No. 1, 21-38 (1958).

The influence of the ratio of the hardness of the oxide film to that of the metal on the response to pressure welding was studied. The deformation required to give an effective weld was determined for a wide range of metals and the effect of rate of loading was investigated. The rate of oxidation of scratch-brushed surfaces of indium, aluminum, zinc, cadmium, copper, tin and lead was determined at room temperature by means of a semi-microchemical balance and autographic recording. The significant factor in pressure welding is the hardness of the metal just before welding. Thus the hardness ratio is a good indication of weldability for metals which work-harden only slightly and have thick (>50Å.) oxide films. In the alloys which work-harden readily, elastic recovery assumes greater importance and there is less plastic yielding, so that the film is not broken up so easily. Suppression of work-hardening by high strain rates was not successful, but suitable alloying resulted in an improvement of weldability. Very thin films are not a serious hindrance to welding, e.g. on lead, cadmium and mercury at —93 C. Grease on scratch-brushed tin surfaces inhibits welding. Aluminum oxide at the interface reduces weld strength.—MA. 15651

3.7.3, 3.2.2

Hydrogen Contents of Mild and Alloy Steel Weld Deposits with Some Reference to the Effects of Hydrogen Embrittlement in Welded Joints. P. D. Blake. *Brit. Welding J.*, **4**, No. 3, 146-154 (1957) March.

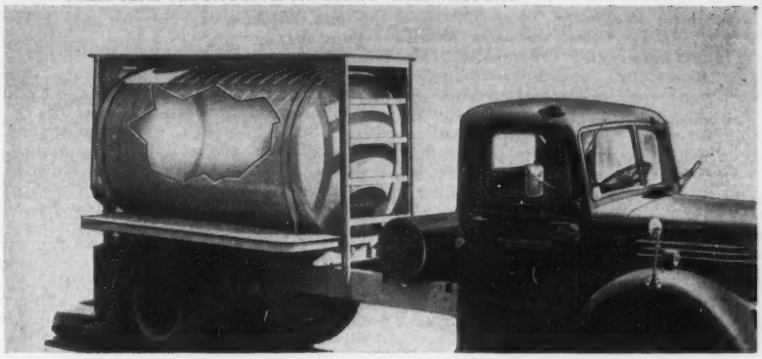
Data on hydrogen content of welds laid down by various classes of mild steel electrodes (cellulose, titania, oxide/silicate iron-manganese, lime-fluoride) to B.S. 1719 and other types of alloy electrodes (18-8, 25 chromium steel, 79 chromium-21 nickel). Data are also given on effect, on hydrogen content of deposit, of drying electrodes immediately prior to welding. Results show that evolution of hydrogen from weld metal at progressively higher temperatures is of non-continuous type. Hydrogen embrittlement is discussed; theory is put forth that four different mechanisms can apply, according to conditions under which cracking occurs. Tables.—INCO. 14113

3.7.3, 4.3.2, 6.2.5, 3.5.8

Trans-Crystalline Corrosion in Sulfuric Acid of Stable Austenitic Acid-Resistant Steels and Welding Joints in a State of Compressive Deformation. (In Russian.) Avtomaticheskaya Svarka (*Automatic Welding*), **10**, 46-50 (1957) Jan.-Feb.

Describes a thermal treatment method as a preventive measure against corrosion.—BTR. 14210

A CHEMISEAL FLUOROCARBON LINING CASE HISTORY



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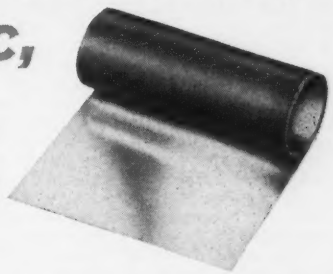

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3.7.3, 6.2.5, 7.2

Austenitic Welds in Type 502 Steel Piping. H. G. GEERLINGS AND W. P. KERKHOF. *Lastechniek* (Hague), **22,** 176-181 (1956) Nov. Abstract: *Welding J.*, **36,** No. 3, 119s (1957) March.

Discusses hard heat-affected zone, difference in coefficient of expansion between weld and pipe, change in structure of weld in service and tendency of weld metal to crack, as difficulties encountered in welding Type 502 piping with 25-20 chromium-nickel electrodes. Cases where cracking was due to sigma formation are considered. Austenitic welded Type 502 piping can be used at service temperatures up to 950 F, according to chart of permissible stresses for various temperatures.—INCO. 13989

3.7.3, 8.9.5, 6.4.2

How the Welding of Aluminium Alloys Has Influenced Their Use in Shipbuilding. H. E. Jaeger. Rev. Soudure, **13,** No. 3, 151-162 (1957).

Shipbuilding applications are listed for aluminum alloys. Requirements are compared with those for aircraft construction, and approximate compositions are tabulated. Good corrosion-resistance is generally accompanied by satisfactory weldability. Effects of some alloying elements on corrosion-resistance are noted, the aluminum-magnesium group containing up to 4.7 percent magnesium resisting both sea-water and inter-crystalline attack. Riveting is considered inferior to welding for light-alloy marine work, as cold-riveting sets up shear stresses and hot riveting is difficult on account of the high thermal conductivity and low shrinkage of the materials. The mechanical properties of multi-run Argonarc-welded joints in Peraluman are compared with those of the unwelded material. The mechanical basis of light-alloy design is reviewed. Composite design (steel hull and light-alloy superstructure) gives the best stress distribution. Proposed thicknesses, moments of inertia and section moduli of various light-alloy structural members, as compared with corresponding parts in steel, are summarized.—MA. 15817

3.7.4, 3.8.4, 6.3.13

Metallographic Manifestations of the Air Oxidation of Tantalum at 750 C. Robert Bakish. J. Electrochem. Soc., **105,** No. 2, 71-74 (1958).

Some crystallographic and structural factors influencing the air oxidation of tantalum are presented. It is suggested that preferential oxidation along (100) planes in <100> direction occurs. Lattice imperfections appear to act as sites for nucleation of the metal-oxide reaction. 12 references.—MA. 15942

3.7.4, 6.4.2, 4.3.2

Study of Intercrystalline Corrosion by Means of Hydrochloric Acid in High-Purity Aluminum and in Aluminum Purified by the Zone Melting Technique. (In French.) F. Montariol. Corrosion et Anticorrosion, **6,** No. 3, 101-106 (1958) March.

Effect of metal purity on the preferential corrosion of the grain boundaries of very pure aluminum in hydrochloric acid. Corrosion is slower with very pure metal. Certain additions to the reagent accelerate the process.—BTR. 15706

3.7.4, 2.3.6, 6.4.2

The Spiral Patterns Observed on the Surfaces of Aluminum Crystals. Seiichi Karashima. Mem. Inst. Sci. and Ind. Reseach, Osaka Univ., **14,** 79-90 (1957); Chem. Absts., **52,** No. 3, 1883 (1958) Feb. 10.

High-purity aluminum (99.991%) single-crystal plates 80 mm long, 13 mm wide and 1.5 mm thick, are made by the strain-annealing method. These samples are then extended approximately 8% and polished electrolytically to approximately 0.5 mm in thickness. Under microscopic examination, to investigate inhomogeneities of deformations by the shapes of the etch pits, many interesting spiral patterns are observed on the surface. In order to determine their origin, the specimens of the same purity aluminum single crystals or consisting of several coarse grains are electropolished under various current densities. Thus similar spiral patterns are obtained on aluminum crystal surfaces during electrolytic polishing at high current density. It is concluded that these figures represent growth patterns of aluminum oxide deposited on the surface. These patterns are observed microscopically because of their grooved structures formed by selective etching action. This observation is made clear by electron micrographs and etching experiments of the specimens with these figures on the surface. These patterns are illustrated. —ALL. 15901

3.7.4, 6.3.19

The Formation of Polycrystalline Lattice Planes (in Zinc). Barbara Borges and Will Kleber. Z. Metallkunde, **48,** No. 10, 557-558 (1957).

When 99.995% zinc single crystals were produced by the Bridgman technique, small droplets formed in a spherical appendix of the tube. After solidification these droplets were found to consist of polycrystalline aggregates bounded by well-developed basal (0001) planes, which did not tarnish during a week's exposure to the atmosphere.—MA. 16134

3.8 Miscellaneous Principles

3.8.2, 6.3.11, 3.6.5

Electrochemical Behavior of Platinum. Equilibrium Diagram Voltage Versus pH of the System Platinum-Water at 25 C. (In French.) J. Van Muylder, N. de Zoubov, and M. Pourbaix. Centre Belge d'Etude de la Corrosion, Rapport Technique No. 63, 11 pp., March, 1958.

Platinum and different platinum compounds in the solid or dissolved state were studied. Free enthalpies. Stability and corrosion of platinum formation of oxides and hydroxides of platinum. 2 plates.—BTR. 16096

3.8.3, 6.4.2

Studies of the Anodic Behavior of Aluminum. Pt. II. Coulometry of Barrier Layer Production. Robert C. Plumb. Cambridge University, England. J. Electrochem. Soc., **105,** 498-502 (1958).

In order to avoid solvent action by the electrolyte during anodic oxidation of aluminum the electrolyte must have a high buffering capacity. With such an electrolyte the conversion of aluminum to aluminum oxide is essentially quantitative. The film thickness is proportional to the voltage drop across the film, and the proportionality constant does not depend on the length of time which the voltage is left applied. The film contains as an essential part of its structure, a quantity of the anion from the electrolyte. The film appears to be nonstoichiometric, having excess aluminum during formation, the amount of excess decreasing with increased length of anodizing time. The coulombic transfer during film formation is quantitatively accounted for during formation by oxidation of aluminum to the trivalent state and liberation of oxygen gas. After the film growth has ceased, the current can be only partially accounted for by oxygen evolution, but it is thought that this is because of difficulty in nucleating oxygen bubbles. 16091

3.8.3, 6.3.19

The Anodic Oxidation of Zinc and Zinc-Tin Alloys at Very Low Current Density. S. E. S. El Wakkad et al. J. Electrochem. Soc., **105,** No. 1, 47-51 (1958) Jan.

Experiments on the anodic oxidation of zinc and zinc-tin alloys at a very low current density were carried out in solutions of various pH values using the direct potentiometric method. With zinc the primary oxidation product was a film of zinc oxide or hydroxide less than one molecule thick. With zinc-tin alloys, zinc hydroxide, stannous and stannic hydroxides were formed before hydrogen evolution. Smaller quantities of electricity were required to passivate the alloys than the pure zinc. Passivation of the aloys in 0.1N nitric acid is said to be due to a chemical process, not an electrochemical one. 5 figures.—ZDA. 15599

3.8.3, 6.3.2

The Anodic Oxidation of Cadmium. Pt. I. Mechanism of Film Formation. P. E. Lake and E. J. Casey. J. Electrochem. Soc., **105,** No. 1, 52-57 (1958) Jan.

Cadmium oxidizes anodically in potassium hydroxide and potassium carbonate solutions with the formation of passive films of cadmium oxide which are later converted to the hydroxide or the carbonate respectively.—ZDA. 15596

3.8.3, 3.6.5, 6.3.13

The Static Electrode Potential Behavior of Tantalum and Its Behavior on Anodic Polarisation. (In English.) E. M. Khairy and M. Kamal Hussein. Univ. of Cairo. Bull. Chem. Soc. Japan, **30,** 944-949 (1957) Dec.

A study of tantalum static electrode potential is of importance for obtaining insight into its mode of oxidation leading to the observed resistivity. For this purpose, the electrode potential of tantalum was measured in buffer solutions covering the pH range 1 to 12 and initially free from its ions. Anodic polarization experiments by the repetitive oscillographic method were utilized in buffer solutions and in other different media.—NSA. 15808

3.8.3, 3.7.3, 6.2.5

Passivity of Stainless Steel in Sulphuric Acid: Influence of Heat-Treatment Between 1050 C and 650 C and Cold-Working. (In French.) C. Carius. Paper before CEBELCOR, Brussels, Nov., 1957. Metaux Corrosion-Industries, **33,** 6-29 (1958) Jan.

Detailed study of passivity of steels containing 0.05-0.08 carbon, 18-18.5 chromium, 9.7-15% nickel, 18/8-type chromium-nickel steels and 18/8/2 chromium-nickel-molybdenum steel in water and aqueous sulfuric acid solutions and in calcium and potassium chloride solutions. Discussion of pitting is included. —INCO. 16057

3.8.3, 6.3.15

Passivity of Titanium in Hydrochloric Acid Solution. R. Otsuka. Tokyo, Scientific Research Institute, J., **51**, 73-74 (1957) June.

The passivity of titanium in hydrochloric acid solutions is attributed to hydride formation which is passivated on exposure to air. The passive state thus obtained was unstable but could be insured by immersion in nitric acid solution. Potassium chloride solution was also helpful. The corrosion loss difference between passivated and unpassivated specimens corresponds to the times required to destroy the passivity of titanium and titanium hydride. The passive state on the latter is the more stable.—MR. 15907

3.8.4, 3.6.5, 3.8.3

Chemical Evidence for Oxide Films on Platinum Electrometric Electrodes. Fred C. Anson and James J. Lingane. J. Amer. Chem. Soc., **79**, No. 19, 4901-4904 (1957).

Chronopotentiometric and analytical studies confirmed that oxidation of platinum electrodes is a PtO/PtO_2 film-formation, rather than an oxygen-adsorption process, and that the films inhibit oxidation of other substances at the electrode. The ratio of the two oxides is approximately the same, irrespective of whether the oxidation is a result of anodic polarization or of chemical action.—MA. 16126

3.8.4, 3.6.8

Stoichiometric Numbers and Hydrogen Overpotential. A. C. Makrides. J. Electrochem. Soc., **104**, No. 11, 677-681 (1957).

Discusses the stoichiometric number of a reaction mechanism and greatly extends the definition. Some objections are mentioned to previous definitions. The definition given extrapolates from reaction rates near equilibrium and is independent of any reaction-rate theory. It has the advantage that it can be used in the case of two or more competing reaction mechanisms. The expected stoichiometric numbers are derived for various possible mechanisms for electrochemical hydrogen evolution. A statement of the Tafel slope and stoichiometric number for a slow discrarge and rapid combination does not uniquely determine this reaction as the mechanism of hydrogen evolution. 15 references. —MA. 15873

3.8.4, 6.2.2

On the Lag Interval in Hydrogen Reduction of Products of Oxidation of Metallic Iron at High Temperatures. (In Italian.) Agostino Bargone and Vittorio Gottardi. Tecnica Italiana, **12**, 449-455 (1957) Oct.-Nov.

Review of research on oxidation of iron and hydrogen reduction of resultant products; experimental observations which explain lag period observed during reduction. Lag is related to composition and morphology of the oxides and to temperature and duration of oxidation process.—MR. 16136

3.8.4, 6.3.14

An Examination of Oxide Films on Tin and Tinplate. S. C. Britton and K. Bright. Metallurgia, **56**, No. 336, 163-168 (1957).

Rates of growth of oxide films were studied by the establishment of combined oxygen by controlled cathodic reduction in a buffered solution and by separation of films from the metal by dissolution of the metal in mercury. Oxide films were produced both by heating in air and by a variety of passivation treatments. Using tinplate, the oxygen content of the films was determined by cathodic reduction and by wt.-gain measurements after heating at 200, 210 and 220 C; good agreement was found. X-ray examination of the film proved it to be stannous oxide. Tin specimens were heated in dry air at 30-210 C and it was shown that the amount of oxide formed α log(time) at 30 C and α (time)$^{1/2}$ at temperature greater than 180 C. Films formed at more than 170 C are stannous oxide, less than 170 C they are probably amorphous. The addition of 0.1% indium or phosphorus considerably reduces, and the presence of moisture increases, the oxidation rate. Films formed from chromate solution contain chromium. 12 references.—MA. 15761

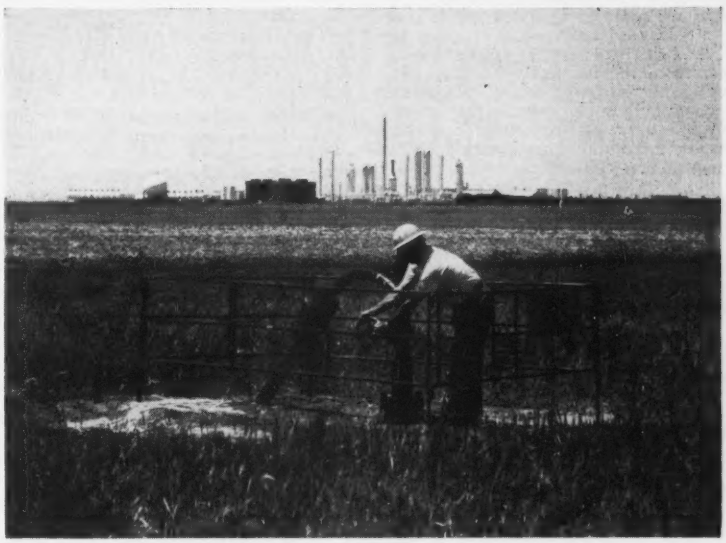
3.8.4, 6.3.15

Study of the Oxidation of Titanium and Its Alloys in Air. (In Japanese.) Shiro Ogawa, Denjiro Watanabe, Yo Takeuchi and Rokuro Kawanishi. J. Japan Inst. Metals., **21**, No. 6, 410-414 (1957).

The alloys contained 3-10% aluminum and 0.3-1.5% silicon, and the weight gain and electron-diffraction patterns of the surface films formed were studied. The growth law was not purely parabolic. Only titanium dioxide was observed, mainly of the rutile-type with some anatase-type at the lower temperature. At 700-900 C the rutile crystals showed two types of selective orientation. 11 references.—MA. 15918

3.8.4, 6.3.20, 3.5.9

Reaction Kinetics of Zirconium and Zircaloy-2 in Dry Air at Elevated Temperatures. L. F. Kendall, R. G. Wheeler



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and S. H. Bush. Nuclear Science and Eng., **3**, No. 2, 171-185 (1958) Feb.

Oxidation rates of sponge zirconium and Zircaloy-2 were measured at 500, 600 and 700 C in dry air, using specimens typical of the products presently available for reactor structural service. Following variables were studied for each metal: temperature, shape, amount of cold work and gas flow rate; only temperature was significant. At any given temperature, reaction proceeds in two stages; rate of reaction first decreases with exposure time; after prolonged exposure, rate becomes linear function of time. Temperature dependence of rate constants is described by Arrhenius equation, which may be used to extrapolate data to other temperatures. Extrapolations show that oxidation rates are so slow that several years service may be expected for zirconium and Zircaloy-2 structures at temperatures below 400 C. Measurement of dimensional changes revealed much scatter, sensitivity to specimen geometry and surface dependence. Oxide formed on zirconium is under compressive stress and under certain conditions metal will flow to relieve this stress. Photomicrographs compare corrosion films on zirconium and Zircaloy-2. Tables, graphs.—INCO. 15635

3.8.4, 4.6.2, 6.2.2

Constitution and Morphological Study of the Film of Superficial Iron Oxidation in Steam at High Temperatures. (In French.) Jean Paidassi and David Fuller. Comptes Rendus, **246**, 604-607 (1958) January 27.

Investigations between 700 and 1250 C showed that the adhesion of the film to the metal is very different according to the oxidizing atmosphere used. The film is made of one layer of ferrous oxide only, except between 900 and 950 C where there is a discontinuous external layer of magnetite.—BTR. 15475

3.8.4, 3.7.2, 6.4.2, 3.5.9

Oxidation of an Aluminum-3 Per Cent Magnesium Alloy in the Temperature Range 200-550 C. W. W. Smeltzer. J. Electrochem. Soc., **105**, No. 2, 67-71 (1958) Feb.

A study of the oxidation and magnesium evaporation kinetics has been carried out on an aluminum-3% magnesium alloy. A metallographic examination of the surface oxide films produced was included. The alloy was oxidation resistant to 200 C. At temperatures greater than 350 C. the oxidation rate was initially inxersely proportional to the oxide film thickness, transforming to a constant rate for long exposures. Selective oxidation of magnesium caused formation of aluminum inclusions in the surface oxide. These inclusions imparted a black discoloration to the alloy surface. Magnesium evaporated from the alloy in a vacuum at temperatures greater than 350 C. after an induction period, the duration of which was dependent on the temperature. A comparison of the magnesium evaporation and alloy oxidation rates demonstrated that the oxide film offered resistance to oxidation of the metal. (auth)—ALL. 15481

3.8.4, 6.3.20, 4.3.2

A Reaction Rate Study of the Corrosion of Low-Hafnium Zirconium in Aqueous Hydrofluoric Acid Solutions. T. Smith and G. R. Hill. Paper before Electrochem. Soc., Cleveland, Sept. 30-Oct. 4, 1956. J. Electrochem. Soc., **105**, No. 3, 117-121 (1958) March.

Zirconium dissolves readily in hydrofluoric acid solutions leaving a smooth unpitted surface. Rate of dissolution of zirconium in aqueous hydrofluoric acid solutions was studied over a temperature range of 7-78 C using a radioactive tracer Zr^{95}. Rate was found to be directly proportional to un-ionized hydrofluoric acid concentration. In order to control equilibrium concentrations of un-ionized hydrofluoric acid, HF^{-}_{3}, H^{+}, and F^{-} ions, most of the solutions contained another component such as hydrochloric acid, nitric acid, potassium fluoride or perchloric acid. For a given un-ionized hydrofluoric acid concentration, rate was independent of HF^{-}_{3}, H^{+}, F^{-}, Cl^{-}, NO^{-}_{3}, K^{+}, or ClO^{-}_{4} concentrations over a wide range. Graphs, diagrams.—INCO. 15591

3.8.4, 5.9.4, 6.4.2

The Structure of Anodic Oxide Films on Aluminum. (In Italian.) M. Paganelli, Alluminio, **27**, No. 1, 3-12 (1958) January.

Anodic films produced in sulfuric acid, chromic acid and phosphoric acid electrolytes were subjected to an electron microscopic investigation, and the dimensions of the elements constituting the cells of a porous anodic film have been measured. The findings were in good agreement with the views of earlier investigators. It is noted that structures built up by the various anodizing processes currently used show slightly different characteristics which may account for their specific optical properties. A critical examination of recent work done in this field is presented. —ALL. 15469

3.8.4

Colloidal Interpretation of Corrosion and Inhibition Phenomena. (In French.) Jean Frasch. Corrosion et Anticorrosion, **6**, No. 1, 9-14 (1958) Jan.

Colloids of metals and their hydroxides. Electrical charge of amphoteric colloids. Corrosion and protection of metals producing such colloids. Electrochemical theories of iron corrosion and colloids.—BTR. 15436

3.8.4, 5.8.3, 4.3.2, 6.3.19

Velocity of Dissolution of Zinc and Influence of Inhibitors Upon Decrease of Corrosive Attack in Aqueous Agents. (In German.) T. Markovic, Z. Dugi and Lj. Rubinic. Werkstoffe u. Korrosion, **9**, 17-20 (1958) Jan.

Behavior of 99.9% zinc in aqueous acid and alkaline solutions represented in corrosion current-pH diagram. Equation for inhibition of dissolution of zinc in aqueous solutions of nitric and sulfuric acid containing sodium dichromate. 20 references.—MR. 15588

3.8.4, 6.2.2, 3.5.9

The Kinetics of the Air Oxidation of Iron in the Range 700-1250 C. J. Paidassi. Acta Metallurgica, **6**, No. 3, 184-194 (1958) March.

Work on superficial air oxidation of iron. In range of 700-1250 C, the scale is always composed of 3 continuous and compact layers of ferric oxide, ferrous ferric oxide and ferrous oxide. Under isothermal oxidation conditions, growth law of these layers is parabolic, except during initial instable stage. In same range of temperatures, relative thicknesses of ferric oxide, ferrous ferric oxide and ferrous oxide layers versus total thickness of scale remain practically the same and are respectively 1, 4 and 95%. Heat of activation of 40,500 cal per mole, illustrates both growth process of one or another of these layers and of the whole scale. Results are concerned exclusively with scales strongly adherent to the support under experimental temperature since appearance of a discontinuity, e.g. at metal oxide interface, during oxidation modifies completely the constitution of the scale. Photomicrographs, graphs. —INCO. 15589

3.8.4

Oxidation of Metals. Walter W. Smeltzer and Lyndon H. Everett. Ind. and Eng. Chem., **50**, No. 3, Part II 496-502 (1958) March.

The literature concerning oxidation of metals is reviewed and an extensive bibliography included. The major contribution of studies on the common base metals has been a critical assessment of prevalent theories on oxidation mechanisms and the role of structural properties of surface oxide layers. The elucidation of oxidation mechanisms by studies on thin oxide films and of ion currents during electrolytic formation of oxide layers in aqueous solutions has been of particular interest. Investigations on the properties of metal oxides have covered a broad field: structures, metal ion diffusion coefficients, electrical conductivity, capacitance and magnetic effects. Also, determinations of the thermodynamic properties of binary and ternary metal-oxygen systems have been carried out.—ALL. 15655

4. CORROSIVE ENVIRONMENTS

4.2 Atmospheric

4.2.5

Corrosion in a Marine Atmosphere. (In Italian.) Giampaolo Bolognesi. Rivista di Meccanica, No. 159, 13-16 (1957) April 13.

Special problems of large installations where frequent anticorrosion treatments are costly or otherwise not readily executed; case of specialized installations, such as oil company docks, where marine medium is altered by other substances. Pitting, effect of scale left by rolling operations, corrosion effects of tide and mud, corrosion of welded parts. —MR. 15712

4.2.1, 5.7.4

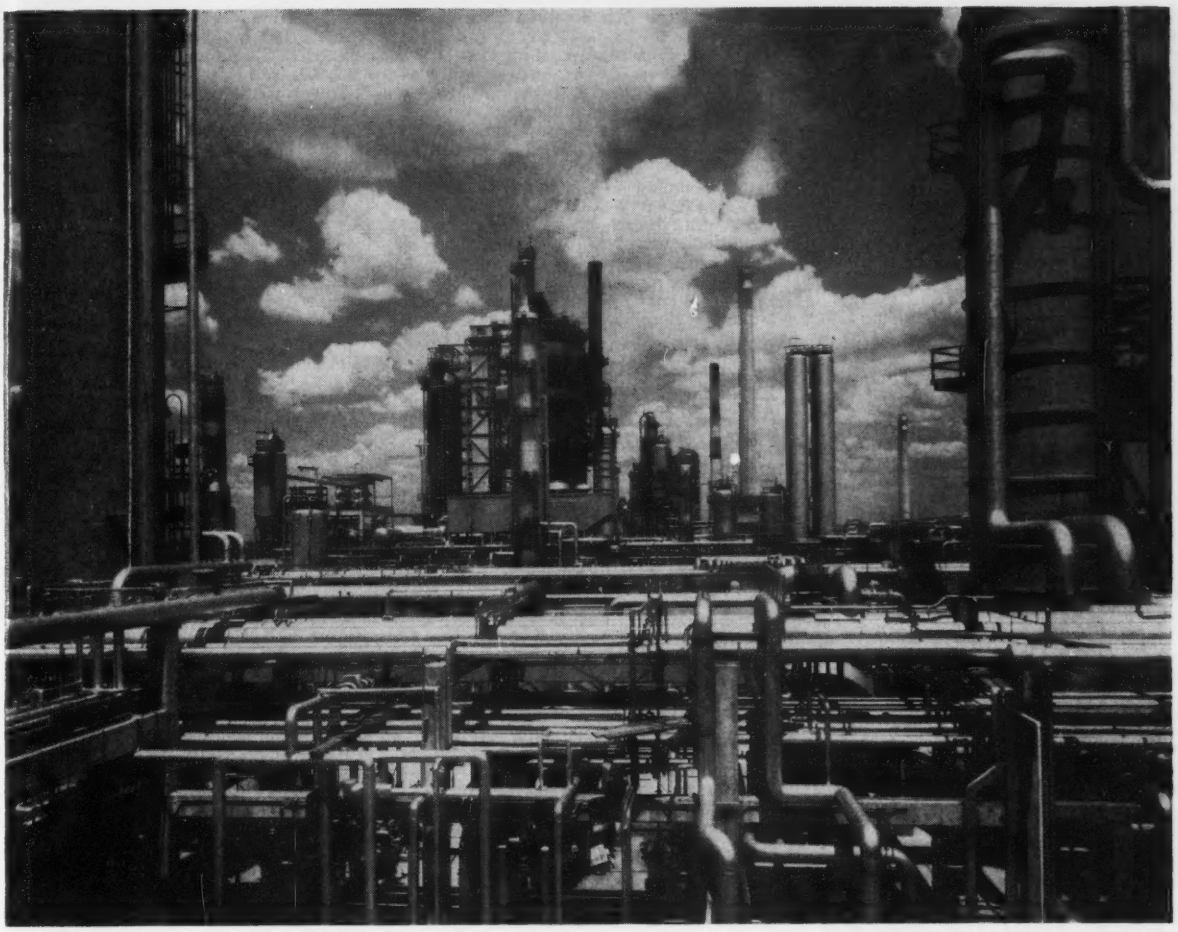
Inhibition of Corrosion by Atmosphere Treatment. (In German.) A. Kutzelnigg. *Werkstoffe u. Korrosion*, **8**, 492-498 (1957) Aug.-Sept.

Survey of corrosive air pollutants and traces, fog and condensed moisture. Air conditions in workshops; critical humidity, methods of dehumidification, mist dispersion, dust removal and detoxication; protection by use of kraft paper for packing purposes. 52 references.—MR. 14966

4.2.7

Corrosion of Metal Surfaces in Tropical Climates. (In German.) O. Marsch. *Werkstoffe u. Korrosion*, **8**, 688 (1957) Nov.

Tropical conditions are simulated by testing products at 40 C and 90% relative humidity. Products made of nonferrous metals or with varnished surfaces are preferred. Electric equipment



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with bright surfaces must be electroplated with copper, nickel, zinc, gold or silver. Sweating of insulating materials causes corrosion.—MR. 15368

4.3 Chemicals, Inorganic

4.3.3, 6.2.5

Corrosion of Stainless Steel by Uranyl Sulfate Solutions Free of Stainless Steel Corrosion Products. A. L. Bacarella. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., CF-56-2-3, Feb. 9, 1956 (Declassified March 16, 1957), 17 pp. Available from Office of Technical Services, Washington, D. C.

Apparatus is described which was used in a study of the corrosion of stainless steel by uranyl sulfate solutions in a system in which the products of corrosion do not accumulate. A schematic drawing of the open ended loop equipment is included. The solution for all experiments was approximately 0.17 molal uranyl sulfate. The experimental conditions for each run and the corrosion results for each are summarized. Results are compared with results obtained in rocking bomb and in static autoclave bomb experiments.—NSA. 16139

4.3.3, 6.3.17

Corrosion Newsletter No. 1. J. L. English. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., CF-51-4-20, April 4, 1951 (Declassified Feb. 14, 1957), 12 pp. Available from Office of Technical Services, Washington, D. C.

A study was made of uranyl sulfate solution stability at 250 C as a function of exposure time in untreated stainless steel systems. It was concluded that total uranium reduction from the uranyl sulfate was nearly 100% complete in 8 hours, that no definite correlation of U^{+4} and total pH vs. total uranium content could be obtained, and that type 347 stainless steel showed a slight weight increase during the first four hours. The effect of Graphitar No. 14 on the stability of uranyl sulfate at 100 C and 250 C was also studied. An average reduction of 9.9% in total uranium content was obtained during 24 hours at 100 C and an 18% reduction in total uranium during 24 hours at 250 C.—NSA. 15768

4.3.2, 6.4.2, 5.8.2

On Some Inhibitors of Aluminum Corrosion. Pt. II. Effect of Tanic Acid on 99.0% Aluminum in 1 Normal Hydrochloric Acid. (In Italian.) G. De Angelis and V. Carunchio. Metallurgia Italiana, **49**, No. 11, 783-792 (1957) Nov. The dissolution of aluminum in 1 normal hydrochloric acid of an electrochemical nature. Tanic acid inhibits the corrosion of 99.0% aluminum by acting on both anodic and cathodic polarization, shifting the reaction potential towards more anodic values.—BTR. 15556

4.3.2, 7.1, 4.3.3

The Resistance of Various Bearing Materials to Chemical Attack by Nitric Acid and Uranyl Nitrate Hexahydrate. Norman D. Groves. General Electric Co., Hanford Atomic Products Operation. U. S. Atomic Energy Commission Pubn., HW-30500, March 11, 1954 (Declassified Feb. 19, 1957), 20 pp. Available from Office of Technical Services, Washington, D. C.

The chemical resistance of various bearing materials, used in Purex submerged pumps, to solutions of nitric acid and uranyl nitrate was studied. Results indicated that Corning glass, boron carbide, titanium, or Chemelac M1-411 exhibited sufficient resistance to chemical attack by 60% nitric acid and by 100% uranyl nitrate solutions at boiling temperatures to warrant their use as bearings in Purex submerged rotating equipment.—NSA. 15797

4.3.2, 7.6.4

Sulfuric-Acid Corrosion in Oil-Fired Boilers and Studies on Sulfur-Trioxide Formation. Donald R. Anderson and Frank P. Manik. Am. Soc. Mechanical Engrs. Paper No. 57-A—199, December, 1957, 12 pp.

Sulfuric-acid corrosion in air heaters and economizers of residual oil-fired boilers. Factors controlling oxidation of sulfur dioxide to sulfur trioxide during combustion process of residual fuels. 35 references.—MR. 15710

4.3.2, 6.3.6, 5.8.3

On the Behavior of Brass in Dilute Solutions of Mineral Acids and Their Mixtures in the Presence as Well as in the Absence of Inhibitors. (In German.) W. Machu and M. G. Fouad. *Werkstoffe u. Korrosion*, **8**, 139-145 (1957) March.

Loss of weight of 60-40 brass in mineral acids or their mixtures, with and without inhibitors, is not additive and does not depend on the accelerating or inhibiting effects of the inhibitors, but on the more or less favorable conditions which prevail for the formation of protective coatings. 15 references.—MR. 13796

4.3.2, 6.4.2, 5.8.2

Some Inhibitors of Aluminum Corrosion. Pt. I. Acid Medium in Presence of Tannic Acid and Rosin. (In Italian.) G. DeAngelis and V. Carunchio. *Metallurgia Italiana*, **49**, 349-354 (1957) May.

Specimens of 99 percent aluminum were immersed in hydrochloric acid. Both tannic acid and rosin showed good inhibiting properties, providing about 99 percent protection in strongest solutions. Study was carried out by measuring loss of weight, reaction potential, surface tension and pH. 31 references. —MR. 14642

5. PREVENTIVE MEASURES

5.2 Cathodic Protection

5.2.1, 4.6.11, 7.5.5

A Trial on Cathodic Protection of Sea-Water Tank. Pt. IV. Testing on the Characteristics of the Coatings Obtained by the Amalgamated Aluminum and Zinc Main Anodes. (In Japanese.) H. Shigeno, Y. Hisamatsu, I. Koizumi and S. Imai. Corrosion Engineering, **6**, No. 4, 214-216 (1957) July.

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The characteristics of the coatings, resulted in the experiment described in the previous report, were examined. The average potential of —980mV (referred to saturated calomel) was observed over the coated surface with the cathodic current of 36 mA/m². Fairly negative potentials were still maintained when the current was stopped for a long time. In spite of the interruption of current for more than 70 days, including the period of exposing the coatings to dry air for a week, the necessary current for complete protection was found to be only 20 mA per sq meter.—JSPS. 15500

5.2.1, 4.6.11, 7.5.5

A Trial on Cathodic Protection of Sea-Water Tanks. Pt. III. On the Electro-Coating by the Combination of Amalgamated Aluminum and High-Purity Zinc Anodes. (In Japanese.) H. Shigeno, Y. Hisamatsu, I. Koizumi and S. Imai. Corrosion Engineering, **6,** No. 4, 210-214 (1957) July.

The third series of experiment was carried out on the electrocoating of a steel tank containing about 100 tons of sea-water. This time, the inner surface fairly rusted was electrocoated with boosting anodes made of aluminum (99.8%) rods superficially amalgamated. An electricity of 62 A•hr was passed on the 1 sq meter of protected area for 72 hours. The coatings obtained were examined and the potential distribution was determined under the protection with high-purity zinc anodes. Then the coated surface was electrocoated again with a current of 91 A•hr per sq meter for 142 hours by means of amalgamated aluminum. The coatings were considerably improved by the additional coating process. The amalgamated aluminum produced an anodic current of 5 mA/cm² and amounted to 1.5--2.0 A•hr per gram of weight loss.—JSPS. 15499

5.2.2, 8.9.3

Anodes Can Protect Your Projects. W. P. Monroe. Sargent and Lundy. Consulting Engr., **10,** No. 2, 80-83 (1958) Feb.

Discusses cathodic protection of underground structures as to galvanic cells, current requirements, economic considerations and location of anodes. Protection of pipelines with coatings and cathodic protection is described. Diagrams, photos.—INCO. 15482

5.2.2, 7.4.2

Cathodic Protection May Boost Corrosion! C. Breckon. Petroleum Refiner, **37,** No. 3, 189-190 (1958) March.

Sacrificial iron anodes are best material to use for protection of copper alloy heat exchanger tubes. High potential available from zinc or magnesium electrodes may give rise to conditions where natural protective films do not form, and such anodes are likely to deprive tubes of beneficial effects of iron corrosion products in the water. Where impressed current system is installed, risk of damage to tubes is minimized by using soluble iron anodes. Photograph shows corrosion in 70/30 copper-nickel tube due to scratch lines near end of tube which created large potential difference between zinc anode and newly-scratched surface and prevented formation of natural protective film.—INCO. 15455

5.2.3, 3.6.9, 4.5.3

Relationship Between the Strength of Cathodic-Protective and Strav Currents Circulating in Underground Cables. (In French.) Stefan Minc and Zbigniew Feldblum. Corrosion et Anticorrosion, **5,** No. 11, 308-314 (1957) Nov.

A steel tube cut into 4 equal lengths, suitably connected to conductors of stray and cathodic currents, was buried in prepared gravel of known humidity and resistivity. The minimum cathodic current ($I_p(o)$) required for prevention of corrosion by stray currents (I_p) of known strength at a given point (x) in the tube is given by $I_p(o) = l^2 \cdot I_p(x)/x(l-x) - \delta\Delta I_p$, where l is the tube length and $\delta\Delta I_p$ is an empirical correction for soil corrosion, the combined action of soil corrosion and stray currents, and the deviation from idea stray-current distribution. The value of $l^2 \cdot I_p(x)/x(l-x)$ is directly measurable.—MA. 15991

5.4 Non-Metallic Coatings and Paints

5.4.2

Glass Coatings. A Good Way to Protect Metals. Charles E. Bullock and Forest Nelson. Materials in Design Engineering, **47,** 106-109 (1958) March.

Chemical and physical properties, performance capabilities and design factors in the use of new glass coatings.—MR. 15620

5.4.2, 5.4.8

Development and Evaluation of Insulating-Type Ceramic Coatings: Part I. Development and Small-Scale Testing. S. Sklarew, C. A. Hauck and A. V. Levy. Marquardt Aircraft Co. for Wright Air Development Center, U. S. Air Force. February, 1958, 98 pp. Available from Office of Technical Services, U. S. Department of Commerce, Washington 25, D. C. (Order PB 131752).

Metal-reinforced refractory coatings effective in the 2000-3000 F range were developed during this search for ceramic coatings capable of protecting alloys used in aircraft and missile propulsion systems. Eight systems were investigated. They were based on sodium silicate, frit, aluminum phosphate binders and a commercial binder designated L-389, all with refractory grain fillers. Most promising for high-stress service in the 3000-F range were the phosphate-bonded compositions. Glass-type bonded compositions such as those containing sodium silicate binder were found to be limited to low-stress service up to 2400 F because of the thermal limitations of the glasses used. Seam welding was evaluated as the best method for attaching reinforcement. Evaluation of the insulating ability of the coatings indicated that temperature reductions up to 1000 F at equilibrium and thermal lag times of about 60 seconds to equilibrium can be obtained through use of reinforced coatings with thicknesses of 0.1 to 0.16 in. and weight of about 0.11 lb per cu in. of coating, including reinforcing media.—OTS. 15657

5.4.3

Modern Tank Linings. A. D. C. Hamilton. Paint J., **7,** No. 53, 438-439, 456 (1957).

Paints based on phenolic resins are used to a considerable extent for the lining of tanks and containers. The dry film, however, is inclined to be brittle and not resistant to alcohol-blended motor fuel or to acids or alkalies. Fairly successful results have been obtained with paints based on vinyl or vinylidene (chloride)/acrylonitrile resins. Isocyanate coatings have also shown promise. Cold-cure epoxide resin coatings give excellent service, being fully resistant to a wide variety of petroleum products, fresh and salt water and dilute acids and alkalies. For all coatings, surface preparation is very important.—RPI. 15800

5.4.3, 7.6.6

Comparative Study of Linings for Sulphite Digesters. J. M. Jopp. Paper before Can. Pulp and Paper Assoc. Pacific Coast Branch, Tech. Sect., Spring Convention, Harrison Hot Springs, B. C., April 25-27, 1957 and Tech. Sect. Summer Mtg., Saranac Inn, N. Y., June 5-7, 1957. Paper Trade J., **141,** No. 30, 28-33, 48 (1957) Sept. 30.

Advantages, disadvantages and cost of carbon steel ceramic brick-lined, carbon brick-lined, stainless steel and stainless steel clad vessels are compared. The first stainless linings were Type 316, but serious corrosion occurred in the domes and later linings included Hastelloy F for these sections. European digesters were all made of a titanium stabilized stainless. Polished Type 316 plates and titanium stabilized stainless show no signs of corrosion. Intergranular corrosion is quite common since factors favorable to such attack result from elevated temperature reached during welding and fabricating of stainless clad plate. Results of study show that it is unlikely that any type of digester will operate for a long period without maintenance. Evidence strongly indicates that a stainless-clad vessel of specifications and design given will produce higher quality and lower cost sulfite dissolving pumps than any of the other three alternatives. Tables, diagrams.—INCO. 15917

5.4.7

Dip Painting and Flow Coating. M. Reeves. Electroplating, **11,** No. 4, 105-109 (1958).

The author comments on dip painting and flow and centrifugal coating processes in the light of personal experience. For the right type of work these processes offer tremendous possibilities for increasing production in the paint shop. Dip painting can readily be incorporated into a conveyorized finishing sequence, particularly where a camelback oven is used. Various types of dip painting machines and conveyorized installations are considered, together with methods for removing surplus coating material. Flow coating is undoubtedly growing in popularity and is applicable to almost every branch of the paint finishing trade, including car components, office equipment, household commodities, metal furniture and service equipment. In a much more limited field, centrifugal coating is a rapid and economical method of applying a single coat of paint or lacquer to small articles. —RPI. 16105

5.4.7

Plastic Properties of Sprayed Aluminum Diffusion Coatings on Steel. S. Gebalski. Prace Inst. Mech., **6,** No. 20, 3-31 (1957).

Sprayed aluminum diffusion coatings are used to prevent oxidation of steel during hot working and heat-treatment. The as-sprayed coating does not show any adhesion, but after diffusion it adheres well and the steel can then be hot or cold worked without cracking or peeling of the coating. The coating consists of a solid solution of aluminum in α-iron with an average maximum aluminum content of 16%; if the steel has

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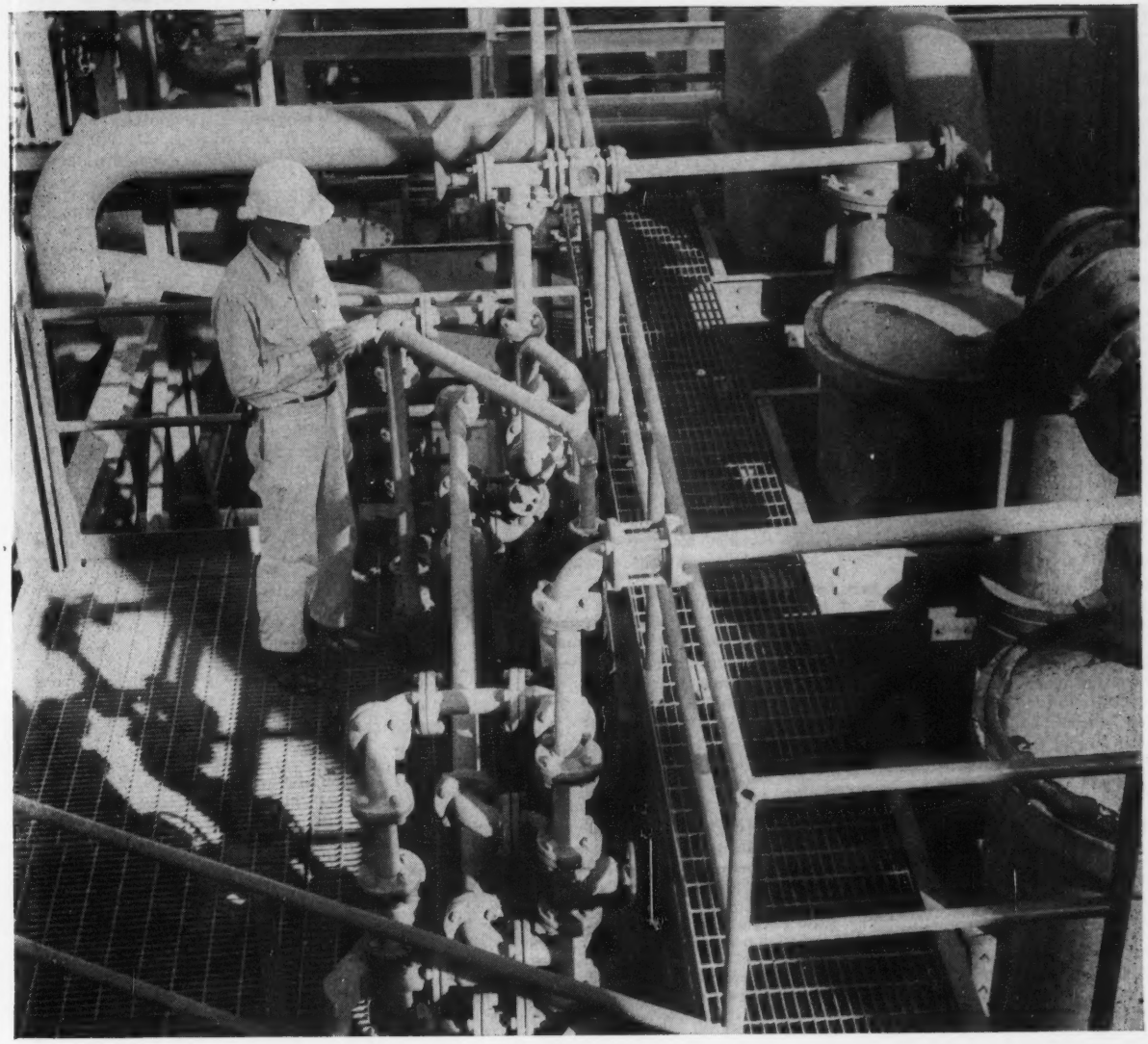

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to be deformed by > 80%, the aluminum content should be decreased. The hot-worked diffusion-coating has good resistance to scaling if it is 0.3 mm thick. This is sufficient for more than 10 working operations in the case of small articles, and 2-3 operations for large parts. The hardness of the diffusion coating should not exceed 400-500 kg/sq mm. Plastic properties can be obtained on heating the component with 3-mm aluminum coatings for 2 hours at over 1000 C. Plasticity ∝ time of heat-treatment and inversely the thickness of the coating and is affected by the shape of the article. The thickness of the coating necessary depends on the number of working operations and reheatings. For up to five reheatings 0.3 mm is necessary, but for small articles worked only once 0.2 mm is sufficient. The coating can be protected from initial oxidation by sodium silicate solution. Forgings need not be preheated to cause diffusion, as the heat of the forging operation will suffice.—ALL. 15839

5.4.8

Plating Wire for Eye Appeal: Survey of Finishes and Processes. R. T. Gore. Wire and Wire Products, **32**, No. 10, 1246, 1291-1295 (1957).

Decorative zinc coatings on wire are best applied continuously with alkaline plating solution of the cyanide type. A basic plating with nickel enhances corrosion-resistance and contributes to a thin, bright, and non-porous coating of zinc, copper, chromium, brass or bronze. To obtain a bright coating of tin heating to temperatures greater than 451 F (233 C) and quenching are necessary after plating.—MA. 15842

5.4.8

Zinc Powder and Zinc Oxide Base Anti-Rust Paints. (In Italian.) Giancarlo Ghisolfi. Pitture e Vernici, **13**, 833-842 (1957) Dec.

Anti-rust protection provided by mixtures of zinc powder and zinc oxide in paints with chemically inert bases and oily bases evaluated by salt spray tests and measurement of electrical potential and voltage in corrosion cells. Films produced by paints studied were applied to inert bases, then investigated for permeability, ease of absorption of solutions, possibilities of reaction with electrolytes. Paints with zinc powder-zinc oxide bases in chemically inert vehicle and having composition very similar to that of a zinc-rich paint develop efficient anti-rust action. 5 references.—MR. 15783

5.4.8

The Use of Epoxide Resin Based Coatings for Corrosion Prevention in a Chemical Refinery. J. L. Francis. Corrosion Prevention and Control, **4**, No. 3, 74-75 (1957) March.

An epoxide resin based primer, pigmented with metallic zinc, followed by one coat of a cold cured epoxide resin based finish is said to give excellent protection to tanks containing tar acids, crude and refined naphtha, and acids and alkalis including concentrated sulfuric acid and strong caustic soda solution.—ZDA. 15786

5.4.10, 5.2.1, 8.9.3

Polyethylene Tape Proves Value. M. E. Parker. Oil Gas J., **55**, No. 2, 133-134 (1957); Chem. Absts., **51**, No. 16, 12478d (1957).

Polyethylene tape used to wrap pipelines requires only about one-third the electric current for cathodic protection of that required for pipe coated with conventional coal-tar coatings, as determined by field tests with 8" welded steel pipelines.—RPI. 15888

5.9 Surface Treatment

5.9.4

A Surface-Treatment Method for the Prevention of Corrosion of Magnesium (Alloy) Castings. G. Nordell and I. Weibull. Corrosion et Anti-Corrosion, **5**, No. 2, 58-60 (1957).

The castings are electrolyzed for 1 hour at 90 V and current density 1.5-2.0 amp/dm², using A.C., in a bath containing (g/l) potassium hydroxide 120, aluminum trihydroxide 30, sodium orthophosphate 35, potassium fluoride 35, potassium manganate 20. The castings are then painted with a varnish containing aluminum powder. The coating is resistant to thermal shock and alloy ZT1 so treated resisted alternate exposure to air/3% sodium chloride for 4 months. Fatigue resistance is greatly reduced.—MA. 15878

5.9.4

Conversion Coatings for Metals. Robert J. Fabian. Materials in Design Eng., **46**, No. 2, 121-136 (1957). Material in Design Eng., Manual No. 140.

Coatings which form an integral part of the basis metal are produced by chemical or electro-chemical means on both ferrous and non-ferrous materials. The production and properties of phosphate, chromate, anodic oxide and special coatings are discussed with illustrations. Coating details include phosphate for aluminum, chromate for aluminum, magnesium and copper, anodized for aluminum and magnesium and oxide and special coatings for copper, brass, zinc, cadmium and titanium.—MA. 15784

5.9.4

The Applications of Bonders. P. de Cerma. Metallurgica Italiana, **49**, No. 5, 377-378 (1957).

Bonders are defined as chemical products that are used to bond organic substances to metals. They are formed in situ by reaction of chemicals with metallic surfaces. Examples are layers of zinc orthophosphate on zinc or aluminum, and aluminum oxide alone or mixed with either (Al, Cr)PO_4 or aluminum orthophosphate on aluminum. These layers are firmly anchored to the base metal; the surface of the layers is rough and porous, and this structure enables them to form firm bonds with other substances, such as oils of all types, greases and lacquers. The bonder layers by themselves are resistance to corrosion; the phosphates in particular have proved useful in this respect.—MA. 15753

6.2 Ferrous Metals and Alloys

6.2.2, 8.8.5

Investigation of Nodular Cast Iron Manufactured in the Springfield Armory Casting Laboratory. O. Lubinkowski. Springfield Armory, U. S. Army Ordnance Corps. May, 1957, 45 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. (Order PB131399).

The advantages of nodular (or ductile) cast iron—good machineability and toughness, excellent resistance to corrosion and high-temperatures—led to this successful effort to prepare thin-wall castings of less than one-half inch thickness. Magnesium silicon, in a little-known application, was used as an inoculant. Results indicated that a high-quality nodular iron can be cast with consistently good results with the magnesium silicon alloy containing copper as the inoculant. For thin-wall castings, formation of predominantly hypereutectic nodules is most suitable; thus the carbon content should be above the eutectic point to attain best conditions for this formation. It was found necessary to superheat the melt prior to inoculation. Addition agents such as ferromanganese, tellurium, Lanceramp, and ferrosilicon were also evaluated in terms of use and importance in producing an iron with fully pearlitic structure and restricted carbide presence.—OTS. 15827

6.2.5, 3.4.8

The Corrosion of Type 347 Stainless Steel in Boiling Digest Liquors. Frederick W. Fink, Walter J. Braun and Oliver M. Stewart. Battelle Memorial inst. U. S. Atomic Energy Comm. Pubn., BMI-1252, Jan. 28, 1958, 40 pp. Available from Office of Technical Services, Washington, D. C.

Corrosion studies indicate that digest liquors presently in use at the Mallinckrodt Uranium Refining Center should not be excessively corrosive to the digesters which are constructed of Type 347 stainless steel. Experimental tests have shown that the digest liquors resulting from some of the more recent sources of uranium concentrates might become corrosive if the fluoride, chloride, or free nitric acid contents of these liquors should reach abnormal levels. If such constituents are present, there are corrective procedures that may be employed to prevent excessive corrosion of the stainless steel. It has been demonstrated that aluminum additions to the digest liquors will reduce the attack in both the liquid and vapor phases when relatively high concentrations of fluoride are involved. Iron additions may be used to some extent to combat corrosion arising from high chloride levels. Type 304 ELC stainless steel appears to have comparable corrosion resistance to Type 347 and Carpenter No. 20 stainless. Haynes Alloy No. 25, which showed a somewhat lower rate of penetration tended to form a loose insoluble corrosion product which may be objectionable in service. (auth.)—NSA. 16047

6.2.5

Iron-Chromium-Aluminum Alloys. J. E. Srawley. U. S. Naval Research Lab. April, 1958, 19 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. (Order PB 131676).

Alloys containing up to 25% chromium and 11% aluminum were produced by vacuum melting and hot worked by forging. When compared with type 310 stainless steel, they were brittle at room and moderately high temperatures and weak above about 1200 F. Nevertheless, the alloys' oxidation resistance overrode these disadvantages in some high-temperature applications, and tests showed that they are resistant to attack by combustion products of residual fuel oils. The excellent hot-working characteristics suggested use in economical production of "dogbone" support members by closed-die forging. Aluminum-containing alloys all had considerably better strength at elevated temperatures than the plain 25% chromium comparison alloy. The effective feature of vacuum induction melting of iron-chromium-aluminum was seen to be simply the exclusion of oxygen, nitrogen, and water vapor during melting and pouring.—OTS. 16097

6.2.5, 4.3.2

Corrosion Behavior of Chromium-Nickel Steels in Concentrated Solution of Sulphuric Acid. V. I. Gintsburg. Khim. Prom., No. 1, 37-42 (1958) Jan.-Feb.

Study of corrosion behavior of steels: St. 2 (low-carbon), Kh 18 N9T (18/9 + titanium), 1 Kh 18N12M2T (18/12 + molybdenum and titanium), and EI530 in concentrated sulfuric acid under various test conditions; test apparatus described. At 85-100 C, marked tendency to pitting of Kh18N9T and comparative immunity of 1Kh18N12M2T were noted. Many external factors influence initiation and development of pitting, in particular oxidizing capacity of solution and rate of flow. First observation of new type of corrosion, described as "nipple" corrosion resulted from prolonged action of sulfuric acid on nickel-chromium steels.—INCO. 15933

6.2.5, 8.9.1, 3.5.9

Evaluation of High Strength Steels for Elevated Temperature Applications in Aircraft. Pt. I. Flat Rolled Products. National Research Council, Materials Advisory Board. April 8, 1958, 90 pp. U. S. Atomic Energy Commission Pubn., Contract DA-36-039-sc-76436.

Two questionnaires, prepared by the Materials Advisory Board but distributed by the Aircraft Industries Association, elicited the information that flat rolled steels required today and in the reasonably foreseeable future for airframe applications should preferably be corrosion resisting and have the following short time mechanical properties.

Service Temperature	Minimum Tensile Strength psi
Room	260,000
800 F	200,000
1000 F	170,000
1100 F or 1200 F	120,000

It was further developed that current methods of presenting creep data are inadequate and that a uniform method of testing new steels for aircraft applications is indicated. All compositions should be readily weldable and have reasonably good machinability consistent with unavoidable high hardness. Steels suitable for high-temperature applications in aircraft and missiles are of six types: hot work steels and martensitic alloy steels, which are not corrosion resisting, and martensitic stainless, semi-austenitic precipitation hardening stainless, cold rolled austenitic stainless and precipitation hardening austenitic stainless steels, all of which have varying degrees of corrosion resistance. Some 27 trade name steels currently in production met one or more of the MAB target values; no one steel met all of them. Of the various steels which met the target values, the hot work steels offer the highest tensile strengths and the best strength/weight ratios up to 1000 F based on short time tensile tests. Martensitic and cold-rolled stainless steels are satisfactory up to about 800 F. No significant break through of the 1100 to 1150 F top usable service temperature for true steels appears likely in the near future although all of the major producers are conducting active research on the subject. (auth)—NSA. 16086

6.2.5, 3.7.3, 4.3.6

The Corrosion of Stainless Steels in Aluminum Nitrate-Sodium Dichromate-Uranyl Nitrate Solution. A. R. Olsen. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., ORNL-195, Nov. 3, 1949 (Declassified March 19, 1957), 8 pp. Available from Office of Technical Services, Washington, D. C.

Various types of welded stainless steel specimens were exposed to a solution simulating in composition that present in a solvent extraction process in the 1200 Area at Oak Ridge National Laboratory. The specimens were exposed singly and in contact with other stainless steels in an attempt to determine the formation rate of corrosion products on or adjacent to welded areas. Stainless steel types 302, 304, 316 and 347 were the materials under consideration for the extraction columns. The results of the tests indicae that none of the stainless steels were subject to serious corrosion attack. Types 302 and 347 showed slightly superior corrosion resistance which was particularly evident in contact couples of these two materials. (auth.)—NSA. 15509

6.2.5, 4.3.2

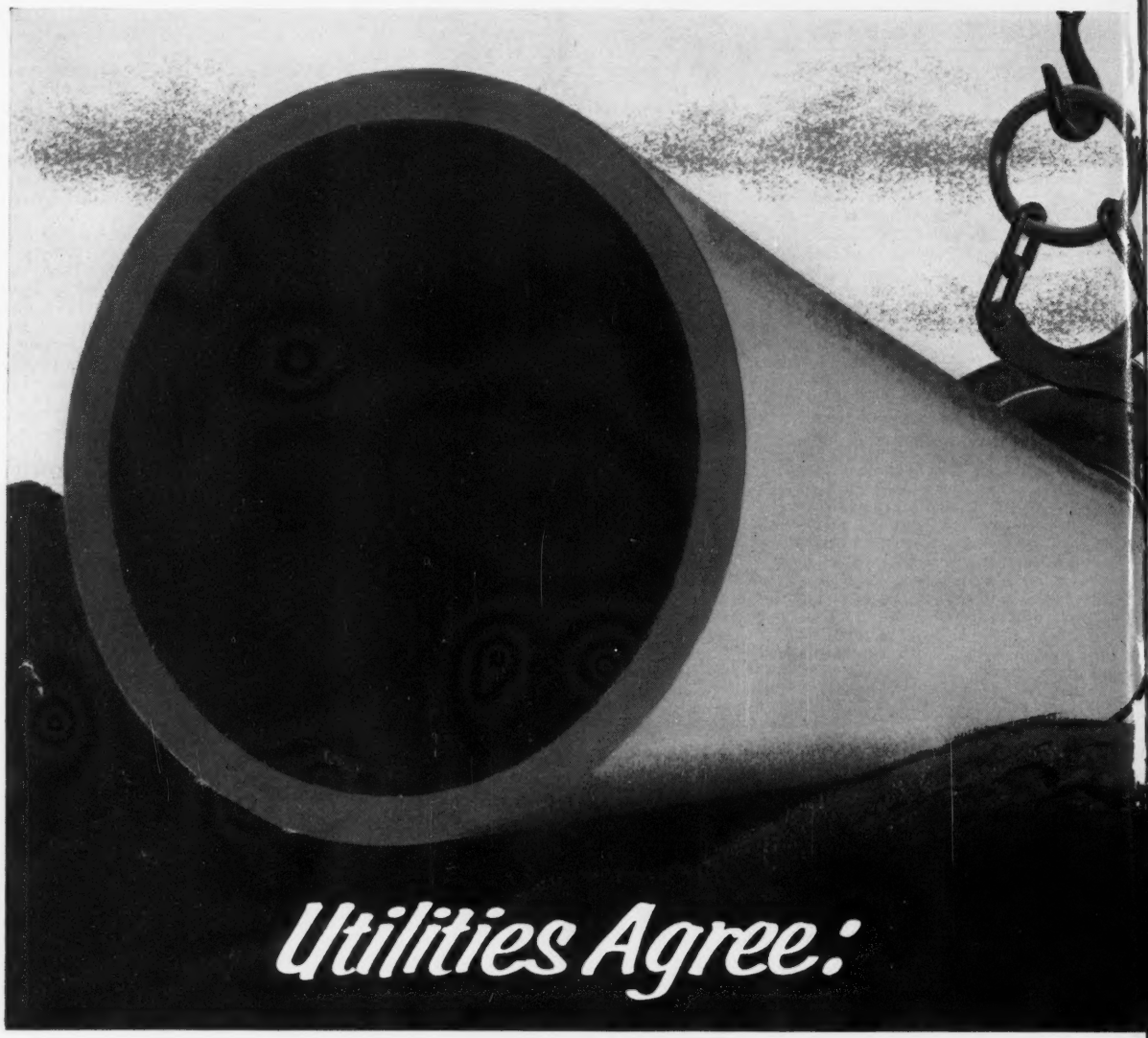
Corrosion of Austenitic Stainless Steel in Mixed Acids. W. W. Koenig. Hanford Works. U. S. Atomic Energy Commission Pubn., HW-17981, June 5, 1950 (Declassified Feb. 19, 1957), 8 pp. Available from Office of Technical Services, Washington, D. C.

An investigation was made of the corrosion rates of several austenitic stainless steels in simulated Redox Process waste storage solution containing hydrofluoric acid, nitric acid and sulfuric acid. Carpenter 20 and T-309 SCb steels showed good resistance to solutions of two concentrations, while T-304 ELC and T-347 showed poor resistance to one concentration and good to fair resistance to the other.—NSA. 15805

6.2.5, 4.3.3, 8.4.5

Studies on Corrosion of 18-8 Stainless Steels in Uranyl Salt Solution. T. Kuroyanagi, N. Shinoda, R. Hasiguti and G. Ito. Paper before Atomic Power Symposium, Jan. 1957. J. Iron and Steel Inst. Japan, **43**, No. 12, 1312-1317 (1957) Dec.

For installation of the solution-type reactor, the authors investigated as to the selection of various types of homemade stainless steel for the manufacture of tanks and pipings of the reactor. It was found that 18-8 stainless steel gave a satisfactory result within a corrosion rate which is permissible for the material to be used for the ordinary reactor. Corrosion resistance of the contact point



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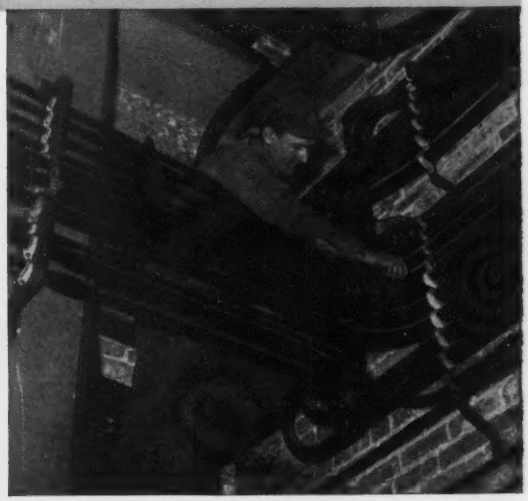

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with uranyl salt solution in the reactor was better if it was immersed in that solution for 1-3 days and subsequently washed beforehand.—INCO. 15803

6.3 Non-Ferrous Metals and Alloys—Heavy

6.3.9

Molybdenum: A Metal with a Great Future. V. Charrin. Chim. et Ind., **78,** No. 4, 423-428 (1957).

The history of molybdenum is outlined. Its ores, extraction and uses in constructional, corrosion-resistant, high-temperature and tool steels are mentioned. Applications in the chemical and allied industries are surveyed.—MA. 15755

6.3.9, 5.3.2, 3.5.9

Molybdenum Alloys and Their Protection Against Oxidation. (In French.) H. Buckle. Recherche Aéronautique, No. 61, 35-52 (1957) Nov.-Dec.

Theoretical and practical problems of use of molybdenum alloys for heat engine parts. Above 500 C protection must be provided by coatings. Those now available can be used at temperatures in excess of 1700 C, but are not entirely satisfactory when subjected to appreciable mechanical stresses. Best protective coatings now known are made of titanium, platinum and chromium, special nickel-iron-cobalt-chromium alloys having unusually low coefficients of expansion; refractory alloys with nickel, iron or cobalt bases, with underlying layer of titanium, platinum or chromium; and alloys containing elements suceptible of forming their own underlying layer, such as $MoSi_2$. Properties of coatings, methods of application and effectiveness of coatings as influenced by application technique. 29 references.—MR. 15718

6.3.9

Coated Molybdenum-Base Alloys. R. T. Begley. S. A. E. Journal, **65,** No. 7, 70-71 (1957).

Molybdenum-base solid-solution alloys, such as molybdenum-0.22% titanium-0.08% zirconium, have 100-hr. rupture stresses up to 65,000 lb/in.^{2} at 1800 F (980 C) and 50,000 at 2000 F (1095 C). The disadvantages of this type of material are a high $\bar{\sigma}$, the difficulty of obtaining high-strength joints, and the lack of oxidation-resistance. Protective coatings must be used if the alloys are to be exposed to oxidizing atmosphere above 1000 F (540 C). Among the more promising coatings are: electrodeposited chromium-nickel, sprayed aluminum-chromium-silicon, clad nickel or nickel-chromium and sprayed or brazed nickel-chromium-boron. The aluminum-chromium-silicon is effective below 2400 F (1315 C), but it is less ductile than the clad or plated coatings. Inconel-clad, molybdenum-3% niobium alloy nozzel vanes were engine-tested at $>$ 2000 F for 30 hr. Two vanes showed evidence of cladding failure. Severe damage was observed in an erosion test. Multilayer coatings (metal + oxide) appear promising to achieve protection without excessive impairment of ductility. $MoSi_2$ has potentialities at very high temperatures.—MA. 15513

6.3.13

Tantalum as a Corrosion Resistant Material. F. G. Cox. Corrosion Prevention and Control, **5,** No. 3, 44-48 (1958) March.

Discusses corrosion resistance of tantalum in acids, alkalies, waters, gases at elevated temperatures, and liquid metals, and reviews application of tantalum in chemical industry. Tabels summarize physical and mechanical properties of tantalum as well as corrosion resistance to long list of chemical agents. Photo shows tantalum lining for hydrochloric acid condenser.—INCO. 15463

6.3.15, 6.3.20

Materials for Corrosion Control. F. W. Fink. Battelle Memorial Inst. Ind. and Eng. Chem., **50,** No. 1, 129A-131A (1958) Jan.

Demand for greater production and efficiency stimulated by pressure of competition and increasing costs, has caused chemical and related industries to look more closely at new materials of construction now becoming available. Both titanium and zirconium are receiving special attention, and research on their corrosion and other properties has been supported by both industry and government. Several thousand feet of titanium tubing and piping will be used to handle sea water at the new nickel plant of the Freeport Sulfur Co. in Cuba. Titanium plating, although not in the production stage, is being studied by Horizons, Inc. Except for greater resistance to alkali, zirconium approaches tantalum in corrosion resistance and recently there has been much interest in its use in chemical plants. Also, zirconium has what is known as a low neutron-capture cross section and because of this, it is used in nuclear applications as a cladding in pressurized-water reactors to provide corrosion resistance for uranium fuel elements. Austenitic stainless steels and Inconel are being studied in dynamic corrosion loops for pressurized-water reactors. Use of inhibitors, additives and cathodic protection in corrosion prevention is discussed. Hydrogen sulfide corrosion and recent studies on factors affecting stress corrosion cracking are considered.—INCO. 15563

6.3.15, 3.7.2, 3.2.2

Development of Improved Titanium Alloys for Application at Elevated Temperatures. B. S. Lement. Manufacturing Laboratories, Inc., for Wright Air Development Center, U. S. Air Force, March, 1958, 73 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. (Order PB 131749).

Marked improvement of the high-temperature strength of titanium results from additions of aluminum. Alloys with aluminum contents higher than six percent, however, may be brittle at room temperature. The nature of this embrittlement was studied in binary alloys containing six to 12 percent aluminum. Changes in bend ductility, hardness, precision length, electrical resistance, lattice parameters and microstructure which occur on solutionizing and aging were determined. It was found that the embrittlement is associated with segregation of aluminum atoms within the alpha solid solution. The segregation takes the form of roughly parallel striations and these appeared to facilitate cracking during bend tests. Embrittlement was also found to be accompanied by decreases in specific volume and electrical resistivity.—OTS. 15661

6.3.15

Titanium. Materials in Design Eng. Manual No. 142. J. L. Everhart. Materials in Design Eng., **46,** No. 5, 149-168 (1957) October.

Provides up-to-date information on all commercial and semi-commercial titanium alloys. Review covers alloys and grades available, mechanical properties, corrosion-resistance; sheet metal forming, forging, casting, machining, grinding, cleaning, finishing, heat treatment, hardening, welding, brazing and soldering; and applications, particularly in aviation, corrosion-resistant equipment and electronics. Numerous tables, graphs, photographs.—INCO. 15570

6.3.15

Alloys of Titanium with Tungsten and Aluminium. (In Russian.) N. T. Gudtsov and I. P. Panchenko. Izvest. Akad. Nauk S.S.S.R., Tekhn. No. 2, 139-143 (1957).

The microstructure, hardness, creep strength and heat-resistance of alloys of titanium with aluminum 3% and tungsten 5, 10, 15% were investigated. The impurities present consisted of carbon ~0.5 and nitrogen ~0.02%. The microstructure of the alloy titanium-3% aluminum consisted of an acicular α-phase and a molecular phase. The alloys with additions of tungsten showed a transformed β-phase and a molecular phase. The chemical analysis of this molecular phase was carbon 10.4-10.6, tungsten 1.75-4.45%, balance titanium with traces of nitrogen. After heat-treatment at 800 C the microstructure of the titanium-3% aluminum alloy consisted of grains of α-phase and molecular phase. The microstructures of alloys with additions of tungsten consisted of a molecular phase only. The heat-treatment decreased the hardness and increased the electrical resistance. The hardness increases with increase of tungsten content. The creep strength of the alloys at 600-650 C is not very high but increases with tungsten content. Aluminum causes a slight increase in oxidation-resistance at 600 C. —MA. 15813

6.3.15, 3.7.3

A Study of the Factors Influencing the Properties of Heat Treatable Titanium Sheet Alloys. R. S. Richards, D. L. Day and H. D. Kessler. Titanium Metals Corp. of America for U. S. Wright Air Development Center, U. S. Air Force, March, 1958, 280 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. (Order PB 131769).

The purpose of this project was to evaluate a number of factors which would influence the properties and subsequent heat-treating response of alloy titanium sheet. The resulting information has valuable practical application in the fields of design and fabrication of the sheet material. Two highly heat-treatable experimental sheet alloys, titanium-2 aluminum-6 molybdenum-2 vanadium and titanium-4 aluminum-3 molybdenum-1 vanadium, were examined. Primary factors studied were effects of cold and warm rolling deformations on mechanical properties. Tensile and bend tests at room and high temperatures were run on materials in the mill annealed, solution, treated, and aged conditions. Studies were also made of aging, strain rate effects, strain distributions in long gauge length tensile specimens, metallographic examinations, dimensional stability measurements, dilatometric measurements during aging, stress-stability tests, modulus measurement, short-time exposure of solution-treated-and-aged material to high temperatures, and a number of other factors.—OTS. 15995

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6.3.15, 3.2.2

Intermediate Temperature Creep and Rupture Behavior of Titanium and Titanium Base Alloys. Part III. Effects of Hot Rolling, Embrittlement and Interstitial Elements. Period covered: March 1, 1955 to December 31, 1955. Jeremy V. Gluck and James W. Freeman. University of Michigan. U. S. Atomic Energy Commission Pubn., Wright Air Development Center, Technical Report 54-112 (Pt. III), Sept. 6, 1957, 115 pp.

Three major factors involved in the properties of titanium alloys at high temperatures were investigated. A limited study was made of the influence of hot-working conditions on creep-rupture properties at 600 to 1000 F. The changes in room-temperature tensile test ductility as a measure of embrittlement during creep testing at 75 to 1000 F were evaluated for a considerable number of specimens of several alloys. A limited study was carried out on the influence of hydrogen content and the hardness of the sponge (interstitial alloying elements) used in making alloys. Creep-rupture properties and embrittlement during stressed exposure were evaluated for temperature of 600 to 1000 F. (auth.)—NSA. 15787

6.3.15, 3.6.5

On the Corrosion-Resistance of Titanium Alloys. Pt. I. The Relation Between the Corrosion-Resistance and the Electrode Potentials of Titanium Alloys. (In Japanese.) Hideo Nishimura and Tsuyoshi Hiramatsu. J. Japan Inst. Metals, **21**, No. 7, 465-469 (1957).

The corrosion ratios and potential-time curves were measured for α and β solid-solution, binary titanium alloys in 20% hydrochloric acid solution. These quantities were also measured for titanium-aluminum-chromium alloys of various compositions in hydrochloric acid, sulfuric acid and oxalic acid solutions. —MA. 15871

6.3.17, 3.7.3

Effects of Metal Purity and Heat-Treatment on the Corrosion of Uranium in Boiling Water. J. E. Draley and J. W. McWhirter. Argonne National Lab. U. S. Atomic Energy Commission Pubn., ANL-5029, April 14, 1953 (Declassified Feb. 12, 1957), 19 pp. Available from Office of Technical Services, Washington, D. C.

Corrosion rates of present reactor-grade uranium were measured in boiling distilled water and were found to have higher values almost by a factor of two than previously reported corrosion rates of uranium. Mallinckrodt biscuit metal showed corrosion rates in the same medium somewhat lower than reactor-grade uranium, and high-purity metal prepared at Argonne National Lab. corroded considerably less rapidly than the biscuit metal. Both reactor-grade metal and biscuit metal showed improvement in corrosion resistance as a result of heating into the β or, more effectively, the γ region and water quenching. In one test, furnace cooling from 1000 C gave the reactor-grade metal its lowest corrosion rate. The high-purity metal showed no such sensitivity to heat treatment but corroded at essentially the same rate regardless of thermal history. The possibility is raised that the reduction of the corrosion rate of less pure uranium by heat treatment is the result of the solubilizing and retention or redistribution of impurities with respect to the matrix phase. The only impurity indicated to be important by the evidence at hand is iron. (auth.)—NSA. 15781

6.3.17, 3.7.3

Heat Treatment and Properties of the Uranium-5 w/o Zirconium Alloy. H. A. Saller, F. A. Rough and A. A. Bauer. Battelle Memorial Inst. U. S. Atomic Energy Commission Pubn., BMI-915, May 17, 1954 (Declassified March 1, 1957), 28 pp. Available from Office of Technical Services, Washington, D. C.

The transformation of the uranium-5 wt% zirconium alloy during heat treatment is an important aspect of the alloy's behavior. Quenching from the gamma-uranium solid-solution region is necessary to minimize corrosion in 100 C water. In this quench condition, the alloy is hard and difficult to fabricate. In larger sections, the stresses built up during quenching can cause warping and rupture. Isothermal heat treatments appear to be the most effective means of avoiding these effects, without altering the good corrosion resistance. (auth.)—NSA. 15427

6.3.20, 4.3.3, 4.7, 3.4.9

Reaction of Zircaloy-2 with Water and with Uranyl Sulfate Fuel Solution. Monthly Progress Rept. No. 2 (for) August 1 through August 31, 1957. Aerojet General Corp. for Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., AGC-AE-37, October 1, 1957, 11 pp. Available from Office of Technical Services, Washington, D. C.

The objective of these investigations is to determine whether the reaction of molten Zircaloy-2 with uranyl sulfate fuel solution (as used in the HRT) is significantly different from the reaction of Zircaloy-2 with water under identical conditions. The scope of the work includes the determination of the violence and extent of the reaction under the following conditions: solid Zircaloy-2 burning in oxygenated water and in oxygenated uranyl sulfate solution at 280 C and 2000 psig; molten Zircaloy-2 spray in water and in uranyl sulfate solution at atmospheric pressure and temperature (using the Aerojet Explosion Dynamometer); molten Zircaloy-2 spray in oxygenated water and in oxygenated uranyl sulfate solution at 280 C and 2000 psig; and molten Zircaloy-2 (in bulk form) in both oxygen-free and oxygenated water at 280 C and 2000 psig, in both oxygen-free and oxygenated uranyl sulfate solution at 280 C and 2000 psig, and in both oxygen-free and oxygenated water vapor at 2000 psig. (auth.)—NSA. 16137

6.3.21, 1.3

The Technology of Thorium. L. L. March, Jr. and I. R. Keeler. Battelle Memorial Inst. U. S. Atomic Energy Commission Pubn., BMI-76, July 18, 1951 (Declassified Feb. 14, 1957), 32 pp. Available from Office of Technical Services, Washington, D. C.

A critical survey is presented of information available at the present time in the project literature and unpublished data on production methods, fabrication techniques, and chemical, physical, and metallurgical properties of thorium metal. 56 references.—NSA. 15862

6.3.20, 3.7.3, 3.4.9

Corrosion Behavior of Zirconium-Base Uranium Alloys. U. Merten and D. C. Belouin. Knolls Atomic Power Lab. U. S. Atomic Energy Commission Pubn., KAPL-1570, June 15, 1956 (Declassified March 28, 1957), 39 pp. Available from Office of Technical Services, Washington, D. C.

The corrosion behavior in high-temperature water of zirconium-base alloys containing 7 to 8 wt% of uranium was studied. A marked dependence of behavior on prior heat treatment was observed. In the annealed condition the alloys lost weight at rates of the order of one mg per dm² per hr of exposure to 360 C water. In the quenched condition the same alloys gained weight consistently, without any visible loss of oxide, for periods up to 4000 hr in length. The use of sponge zirconium or a Zircaloy in place of crystal bar zirconium in preparing the alloy had no noticeable effect on the corrosion resistance of the annealed material, but appeared to increase the corrosion rate observed after quenching. (ath.)—NSA. 15910

6.3.20, 4.3.2

The Corrosion Resistant Properties of Zirconium. Pt. I. F. G. Cox. Corrosion Prevention and Control, **5**, 39-42 (1958) April.

Purity of zirconium affects its corrosion resistance. Low-carbon (0.04%) zirconium metal is attacked by hydrofluoric acid, concentrated sulfuric acid and solutions of ferric chloride and cuppric chloride; and is resistant to hydrochloric acid, phosphoric acid, dilute sulfuric acid and alkalies. Special alloys, developed for high-temperature use with water.—MR. 16054

6.3.17, 4.6.2, 8.4.5

Pressurized Water Reactor Fuel Element Development Program. Lloyd B. Kramer, editor. Westinghouse Electric Corp. U. S. Atomic Energy Commission Pubn., WAPD-M-44, Nov. 24, 1953 (Declassified March 12, 1957), 48 pp. Available from Office of Technical Services, Washington, D. C.

Two classes of high uranium alloys are known to possess superior corrosion resistance to high temperature water. These are the γ-phase stabilized alloys typified by the uranium-molybdenum class and the α-phase alloys of which a 3% niobium in uranium composition is an example. The former type probably possesses adequate corrosion resistance (at least for a 3000 hr core) and can probably be developed so as to exhibit suitable corrosion resistance for even longer periods. It suffers the disadvantage of introducing a large amount of poison into the system since at present 8 to 9 wt% molybdenum is the minimum required. The latter type is more desirable in regard to cross-section but shows insufficient corrosion resistance for the Shippingport Pressurized Water Reactor (PWR) application since the maximum temperature of usefulness is limited at present to 500 F. Both types of alloys are benefited when restrained in claddings. By suitable combination of proper cladding technique and judicious selection of ternary addition elements, it is believed that the prospects for development of a core alloy showing adequate corrosion resistance at an acceptable poison level through defects in the claddings under Pressurized Water Reactor operating conditions are quite good. (auth.)—NSA. 15819

6.3.17, 8.4.5

Uranium and Its Alloys (in Use in Nuclear Reactors). R. W. Nichols. Nuclear Eng., **2**, No. 18, 355-364 (1957).

The physical and mechanical properties of uranium and its alloys relevant to its use as a nuclear fuel in reactors are summarized. Topics discussed in-

clude: anisotropy of physical properties; distortion and swelling induced by thermal cycling through the $\alpha \rightarrow \beta$ transformation (662 C) and the effect of alloying on this; mechanical properties (tensile, impact, creep), including effect of alloying and heat-treatment; compatibility with other materials, e.g. canning metals (aluminum, iron, niobium, tantalum), water, gases (hydrogen, oxygen, carbon dioxide); fabrication (melting, casting, hot and cold working, powder metallurgy, machining, welding); effects of irradiation, viz. irradiation creep (Cottrell effect), swelling due to gases fission product formation after heavy irradiations. The properties of uranium are tabulated.—MA. 15861

6.4 Non-Ferrous Metals and Alloys—Light

6.4.2, 8.9.5

Aluminum in Ships. C. W. Leveau. Kaiser Aluminum & Chemical Sales, Inc. Paper before Soc. Naval Architects and Marine Engrs., Gulf Section. Modern Metals, **13,** No. 7, 42, 44, 46-47 (1957) August.

Use of aluminum in ship construction is widespread in Europe and has been increasing in the U. S. since end of World War II. Due to practical experience and technical developments, aluminum may be regarded as one of the most modern and useful of shipbuilding materials. New alloys with high corrosion resistance and high weld strength include 5154, 5086, 5083 and X5356. In accelerated stress corrosion tests using preformed specimens and also in natural environment tests of over 4 years duration, no stress corrosion failures were obtained in completely unprotected 5083 and 5086 alloys in commercial tempers under severest conditions. Their stress corrosion resistance in marine atmospheres at ambient temperatures is considered excellent. In sea water, common aluminum alloys will initially undergo pitting corrosion. Pitting may begin at a large number of sites and extend into metal to a depth of 2-3 mils. However, subsequent rate of pitting decreases very markedly. This has been called the self-stopping nature of corrosion of aluminum and is probably due largely to formation of protective corrosion products over small pits. Photos, diagrams.—INCO. 15811

6.4.2, 8.4.5, 4.7

S-D-R Progress Report Advances Aluminum as a Barrier Material. American Metal Market, **65,** No. 54, 10 (1958) March 10.

In experiments carried out by researchers of the Nuclear Corporation of America, aluminum sheet was subjected for 16 hours and at 950 F to the effects of a jet of sodium metal. Since the surface showed no signs of corrosion, aluminum is considered as a barrier material between the heavy water moderator and the sodium coolant used in the sodium-deuterium reactor under development.—ALL. 15446

6.4.2, 4.6.2

Attack of Water on Aluminum at High Temperatures. (In German.) P. Lelong and J. Herenguel. Metall, **12,** 176-179 (1958) March.

Corrosion of aluminum investigated by metallographic examination of specimens exposed to water at various temperatures. Three main stages of corrosion are formation of a homogeneous covering layer; intercrystalline effect; and asymmetric point-like corrosion. Increasing temperature accelerates the process.—MR. 15424

6.4.3, 4.6.2

Corrosion of Beryllium in 600 F Water. D. S. Kneppel. Nuclear Metals, Inc. U. S. Atomic Energy Commission Pubn., NMI-1190, September 10, 1957, 21 pp. Available from Office of Technical Services, Washington, D. C.

In a study of certain variables affecting the corrosion resistance of beryllium in 600 F water, the metal had a maximum life of 140 to 160 days before accelerated local attack. Of the various fabrication procedures studied, only quenching from about 1750 F gave any indication of improving corrosion resistance. Among the alloys tried, 0.66 wt % nickel gave similar promise; one such sample accumulated 280 days' exposure to 600 F water, without signs of failure. (auth.)—NSA. 15345

6.4.3

Beryllium. G. L. Miller. Murex, Ltd. *Nuclear Power,* **2,** 438-440a (1957) Oct.

The fabrication of beryllium is summarized. Discussions are included on the consolidation of metal powder, the importance of particle size, corrosion resistance, health hazards, applications and future, beryllium as a structural material, weight saving and the oxide as a nuclear moderator. A data sheet of the physical and mechanical properties of beryllium is included.—NSA. 15372

6.6 Non-Metallic Materials

6.6.5, 5.4.5, 4.3.2

Protecting Concrete Against Corrosion. H. R. Touchin. *Corrosion Technology,* **4,** No. 12, 417-419 (1957) Dec.

Considers corrosive agents that attack concrete and cement and types of surface coatings that can be used for their protection. Most aggressive corrodents are acids; all mineral acids attack concrete rapidly while organic acids vary in intensity of attack. Bitumens and chlorinated rubber paints are limited to specific uses. Introduction of epoxide resins has created possibility of more universal type of surface coating, one which can be used under variety of conditions. Epoxide resins are applied without pretreatment, exhibit outstanding corrosion resistance, prevent mold growth and are easily cleaned.—INCO. 15128

6.6.6, 4.7

Interaction of Uranium and Its Alloys with Ceramic Oxides. H. M. Feder, N. R. Chellew and C. L. Rosen. Argonne National Lab. U. S. Atomic Energy Commission Pubn., ANL-5765, July, 1957, 21 pp. Available from Office of Technical Services, Washington, D. C.

The corrosion of ceramic oxides by molten uranium and its alloys was examined. Dense, high-purity alumina, magnesia, zirconia, beryllia and thoria were studied. The studies included: (a) wettability of ceramics; (b) products of reaction; (c) mechanism of reaction; (d) contamination of molten uranium by crucible material; and (e) the effect of alloying additions on these. (auth).—N.S.A. 14894

6.6.6, 4.3.3

Corrosion of Silicate Glasses by Alkaline Solutions. Part 2. Relationship Between the Composition of Glasses and Their Stability with Respect to the Action of Alkalies. V. S. Molchanov and N. E. Prikhid'ko. *J. Applied Chem., USSR* (Zhurnal Prikladnoi Khimii), **31,** No. 1, 3-7 (1958).

Stability to alkaline solutions was determined for glasses of the type (87—x)% silicon dioxide •x% RO•13% sodium oxide, where R is beryllium, magnesium, calcium, strontium, barium, zinc, cadmium, lead; for double sodio-silicate glasses containing 13-33.3% mole-% sodium oxide; and for titanium-containing flints. 3 tables, 16 references. —ATS. 15170

8. INDUSTRIES

8.4 Group 4

8.4.5, 1.2.5

MTR-HRP Rocking Bomb Experiment ORNL-15. J. R. McWherter. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., CF-55-5-158, May 24, 1955 (Declassified Sept., 1957), 12 pp. Available from Office of Technical Services, Washington, D. C.

A study of the effect of high fission density on corrosion of reactor component materials in contact with homogeneous fuel solution is proposed. A small bomb made of the metal to be tested is partially filled with fuel solution and rocked to permit wetting of all surfaces. The bomb is initially pressurized with oxygen and the decrease of this pressure with time is used to determine the corrosion rate.—NSA. 15902

8.4.5, 3.5.4

Radiation Effects in the Chemical Processing of Fast Oxide Breeder Fuel. Samuel S. Jones. Knolls Atomic Power Lab. U. S. Atomic Energy Commission Pubn., KAPL-M-SSJ-3, May 31, 1956 (Declassified March 12, 1957), 19 pp. Available from Office of Technical Services, Washington, D. C.

In processing spent fuel from the KAPL Plutonium Power Breeder Reactor in the Purex Process the effect of radiation damage becomes prominent. The effect of the radiation on the following process factors is discussed; temperature, gas evolution, viscosity, corrosion, solute oxidation states, solvent deterioration and phase interface behavior.—NSA. 15906

8.4.5, 3.4.3

HRP-CP: Conclusions Corcerning the Predicted Behavior of Insoluble Corrosion and Fission Products in the HRT. P. A. Haas, W. D. Burch, R. W. Horton and R. A. McNees, Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., CF-58-1-97, January 23, 1958, 43 pp. Available from Office of Technical Services, Washington, D. C.

Information available from HRP-CP development studies was summarized and used as a basis for predicting the probable behavior of corrosion and fission product solids in HRT and its associated chemical processing plant. Operation of the chemical plant will probably limit the HRT circulating solids concentration to between 10 and 100 ppm; quantitative predictions are uncertain due to uncertainties concerning the characteristics of reactor solids and the effects of reactor geometries. (auth.)—NSA. 15940

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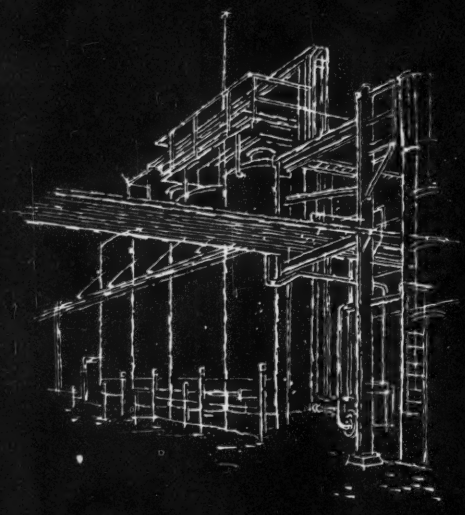

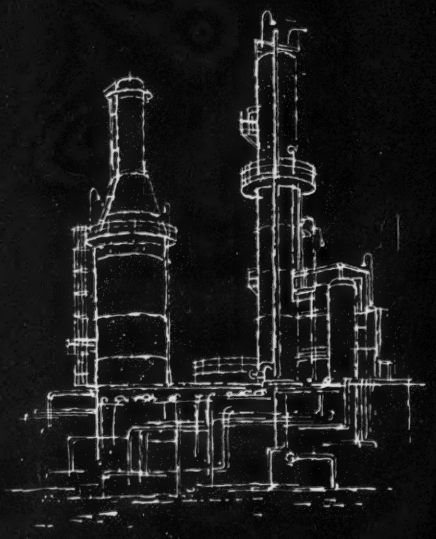

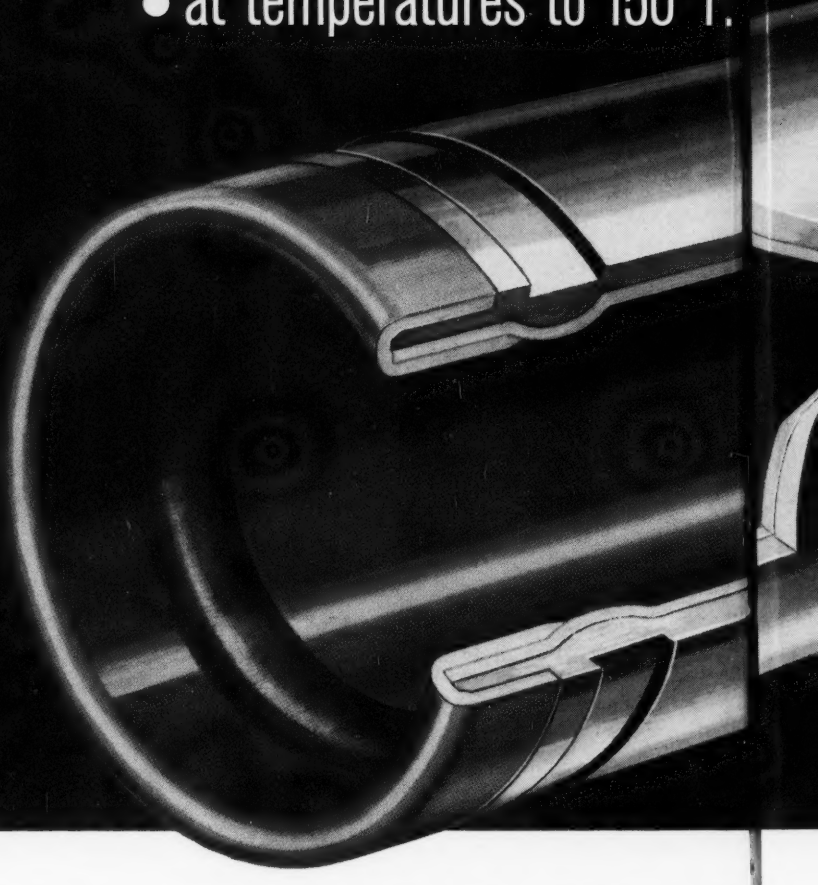

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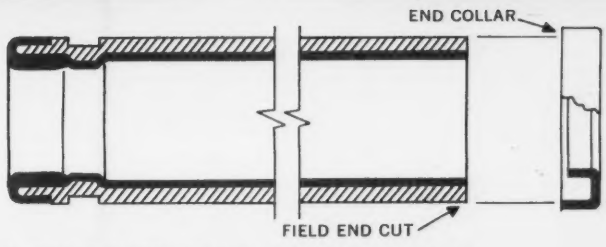

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8.4.5, 4.3.4, 4.3.2

Evaluation of Container Materials for Zircex and Darex Nuclear-Fuel-Recovery Processes. Paul D. Miller, Charles L. Peterson, Earl L. White and Frederick W. Fink. Battelle Memorial Inst. U. S. Atomic Energy Commission Pubn., BMI-1242, December 11, 1957, 44 pp. Available from Office of Technical Services, Washington, D. C.

The selection of a suitable material for the processing equipment is most important in fuel-recovery processes. The problem arises because of the aggressive nature of the reagents that must be used for disintegrating or dissolving some spent fuel elements. In this investigation, corrosion tests were made under conditions simulating those that may occur in several proposed processes in order that candidate materials of construction could be evaluated. No material has been found which would allow both the hydrochlorination and dissolution steps of the vapor-phase Zircex process to be carried out in a single vessel. For a two-vessel process, several materials, such as Inconel, Haynes 25, Type S-816 alloy, Illium R, and the Hastelloys B and C, appear promising for construction of a hydrochlorinator; the dry residue would then be transferred to another vessel, perhaps of titanium or tantalum, for dissolution. In the alternate liquid-phase Zircex process, preliminary tests have given promising results with nickel and nickel-chromium alloys. However, the only metals studied, which withstand the corrosive boiling aluminum-ammonium chloride plus gaseous hydrochloric acid environment with but negligible weight losses have been platinum, gold, tungsten, and molybdenum. A variety of laboratory studies have shown that titanium will apparently withstand any condition likely to occur in the critical areas of the Darex process, including the dissolver and chloride-stripping column. It will undergo a severe attack in hydrochloric acid vapor, but this is easily prevented by the presence of a small amount of nitric acid vapor or oxides of nitrogen. Tantalum is another promising material disclosed by these tests, but its use in the dissolver seems negated because embrittlement was detected under certain conditions. No signs of cracking or other attack have been observed on Type 347 stainless steel specimens exposed at room temperature with various levels of stress for over 6 months to Purex-type acid extraction systems containing chlorides far in excess of the concentrations expected during operations of either the Zircex or Darex head-end processes. (auth.)—NSA. 15453

8.4.5, 5.11

Impact of the Atom on the Design and Manufacture of Pressure Vessels. D. K. Davies. Paper before Atomic Industrial Forum, 4th Annual Conference, New York, October 28-31, 1957, 7 pp. Paper No. 57-AIF-74. Can. Metalworking, **21,** No. 4, 8-10, 12, 14, 16 (1958) April.

Description of characteristic problems associated with design and manufacture of nuclear equipment. When subjected to intense bombardment from high energy neutrons most carbons and low alloy steels undergo radiation damage which results in changes in physical properties of the material. Characteristics of radiation damage are an increase in tensile strength and yield strength of the material, a decrease in ductility and a loss of ability to absorb suddenly applied loads. Effect of radiation damage can therefore be minimized through the use of materials which have low transition temperatures and high initial impact resistance. All reactor vessels must have internal surfaces which are corrosion resistant. In the case of liquid metal cooled reactors, reactor pressure vessel is usually fabricated from austenitic stainless steel. For pressurized water reactors it is more economical and more practical, because of higher coolant pressures used, to fabricate reactor pressure vessel from carbon or low alloy steels. When this is done, internal surface of reactor vessel is made corrosion resistant by the addition of a thin austenitic stainless steel cladding.—INCO. 15681

8.4.5, 6.3.17, 4.7, 3.4.6, 4.4.1

Corrosion of Uranium, Thorium and Uranium Alloys in Sodium and Organics. Harry Pearlman. Atomics International, Div. North American Aviation, Inc. U. S. Atomic Energy Commission Pubn., TID-7546 (p. 565-587), p. 565-87 of Fuel Elements Conference, Paris November 18-23, 1957, 23 pp. (NAA-SR-Memo-2169).

Sodium and sodium-potassium alloys and the polyphenyl class of organic compounds possess certain advantages over water for use as reactor coolants. There are no reactions between uranium and the pure liquid metals; but thermodynamic data predicts that formation of uranium carbide and uranium hydride is possible in reactions with organics and with their pyrolytic and radiolytic products. Available experimental data are reviewed for uranium and uranium alloys exposed both to sodium and sodium-potassium alloys in static and dynamic systems. Effects of irradiation on corrosion rate do not appear to be significant. The observed corrosion rate of uranium and uranium alloys depends very strongly on the impurity, especially oxygen, content of the liquid metal. In capsule type static corrosion experiments, made with liquid metal with oxygen content near 0.01 wt.% (100 ppm), corrosion rates are of the order of 10 mg/cm²-month at temperatures up to 750 C. Uranium dioxide and UO have been identified as corrosion products. In dynamic systems, corrosion rates of more than 600 mg/cm²-month have been observed at 500 C with oxygen content ranging near 0.005 wt.% (50 ppm). Corrosion rates of uranium and uranium alloys in purified biphenyl are of the order of 1 mg/cm²-month at temperatures up to 400 C in static systems. Under dynamic conditions in loop experiments, rates are perhaps five times greater. Formation of uranium carbide, through UH_3 as an inferred intermediary, has been observed in monoisopropyl biphenyl under hydrogen gas pressures of the order of 100 psi. Impurities dissolved in the organic, such as air, oxygen, or water, greatly accelerate the corrosion rate. It is concluded that the corrosion rate of uranium and its alloys in liquid sodium and sodium-potassium is low enough to permit the use of a static liquid metal bond between the fuel and a cladding of some other metal. Exposure of relatively large surfaces of fuel to circulating liquid metal coolant appears not feasible, because of the difficulty of adequate control of oxygen in a large volume liquid metal system. There would be no advantage in reactor performance in direct exposure of unclad fuel to an organic coolant; but, in case of accidental exposure by way of a cladding failure, the corrosive attack would be relatively very slow. (auth.)—NSA. 15884

8.4.5, 6.4.2, 6.2.5, 6.3.20

Corrosion of Fuel Element Materials for a Low Enrichment Power Reactor. A. L. Medin and R. J. Clark, Jr. Alco Products, Inc. U. S. Atomic Energy Commission Pubn., APAE-Memo-44, August 1, 1956 (Declassified July 3, 1957), 175 pp.

To determine their possible use as fuel element material, an evaluation was made of the corrosion characteristics of aluminum, zirconium, stainless steel and their alloys. The corrosion behavior of these metals under conditions that would prevail in a low enriched water cooled and moderated reactor of the APPR is as follows: Zircaloy-2 and 3 are more suitable for reactor application than crystal bar zirconium. The average corrosion rate for zircaloy-2 in 500 F water is 0.05 mg/cm²/mo. Stainless steel was the most corrosion resistant material investigated; its corrosion rate in 500 F water averages 0.04 mg/cm²/mo. Commercial 2S aluminum is unsatisfactory above 400 F. However, when alloyed with nickel or copper, or plated with nickel, corrosion rates are significantly reduced. In 500 F water (pH-7) the corrosion rate of aluminum-1% nickel and aluminum-6% copper alloys is approximately 0.9 mg/cm²/mo (1.5 mils/yr) at a flow rate of 6 fps. Generally, corrosion rates of all these metals vary considerably with changes in temperature, flow rate, pH and other conditions. (auth.)—NSA. 15981

8.4.5, 4.7

Some Studies of the Reaction Between Sodium and Bismuth. R. I. Hawes. United Kingdom Atomic Energy Authority. Research Group. Atomic Energy Research Establishment, Harwell Berks, England, Sept., 1957, 13 pp.

In a circulating fuel, liquid metal fueled reactor, a secondary liquid metal coolant circuit is likely to be used. There is interest in a solution of uranium in bismuth as a fuel and sodium as a secondary coolant. An exothermic reaction occurs between sodium and bismuth leading to a high melting point intermetallic compound. This paper discusses the effects which might occur if a leak occurred in the primary heat exchanger and reports some experiments which were carried out to investigate these effects. (auth.)—NSA. 15802

8.4.5, 6.2.5

Activity from Stainless Steel in Pile Water. Progress Report (for) November 30, 1945. R. B. Briggs, O. Sisman and B. Manowitz. Clinton Labs. U. S. Atomic Energy Commission Pubn., MonN-36, Dec. 11, 1945 (Declassified Feb. 28, 1957), 13 pp. Available from Office of Technical Services, Washington, D. C.

Water was passed through a stainless steel tube installed in the Clinton pile to determine the sources of the activity present in exit water. It was found that most of the activity resulted from the corrosion or recoil of active atoms from the stainless steel and appeared that the recoil mechanism contributed the largest amount. The activity of greatest intensity was that from Mn^{56} derived from the stainless steel. Intensities of activities induced in the chlorine and sodium dissolved in the water were next greatest in that order. (auth.)—NSA. 15522

DURIRON

ANODE DATA SHEET

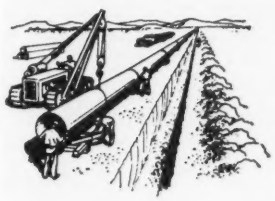
Number 5

Ground Bed

Basic engineering information about DURIRON impressed current anodes for cathodic protection.

Protection of a 30" Steel Water Line

Duriron Anodes Installed Without Backfill



THE PROBLEM: An isolated section of 11,000 feet of 30" steel water line needed cathodic protection. Tests showed the current requirements to be about 40 amperes, but it was planned to replace a section of the adjacent wood-stave line with steel, so extra capacity was desired. Only one site, about 4000 feet from one end of the section, had a power supply available. The ground at this location was a peat bog, with a resistivity of 4000 ohm-centimeters. The water table stood within six inches of the surface, making it impossible to get a dry, clean hole for the installation of anodes.

THE INSTALLATION: Thirty-two Duriron Type D anodes (2" x 60") were used, without backfill (Fig. 1). They were installed by digging through the dry surface soil for about one foot, and then pushing the anodes down until the tops were three feet below the surface (Fig. 2). The calculated resistance for the thirty-two anode group, at twenty foot spacing in two rows, was one ohm, based on previous experience with similar installations. Had it been possible to install anodes in conventional backfill columns, the requirements could have been met with twenty-two anodes. Under the circumstances, the use of bare anodes effected a considerable saving.

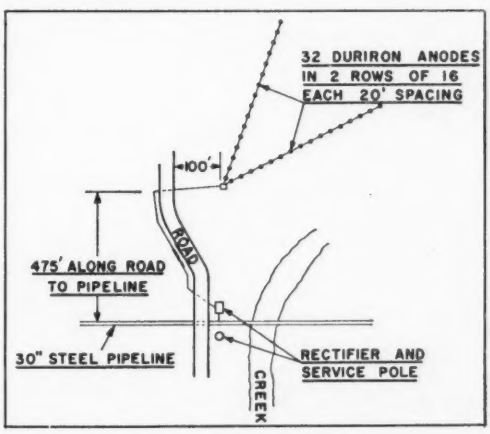


Fig. 1—Illustration of anode spacing

THE PERFORMANCE TO DATE: The anode bed is discharging 60 amperes at a terminal voltage of 59 volts. The entire line section is under protection, the minimum potential being 1.18 volts (referred to a copper-copper sulfate electrode placed over the line). This affords ample margin to handle anticipated additions to the line. It should be noted that the calculated anode resistance $R = .005\rho$ is quite close to the actual resistance of $R = .00491\rho$.

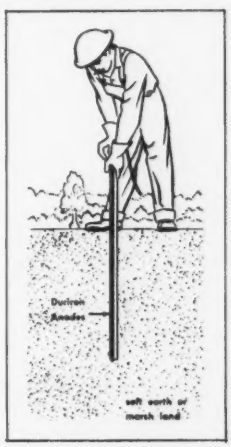


Fig. 2—Method of installing anodes

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